AD-A094 679 NAVAL AIR DEVELOPMENT CENTER WARMINSTER PA AIRCRAFT --ETC F/G 11/4
NATURAL EXPOSURE OF SELECTED GRAPHITE/EPOXY COMPOSITE MATERIAL --ETC(U)
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NATURAL EXPOSURE OF SELECTED GRAPHITE/EPOXY COMPOSITE MATERIAL SYSTEMS

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AIRTASK NO. 62761N ZF61-542-001
Work Unit ZM501

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Prepared for
NAVAL AIR SYSTEMS COMMAND
Department of the Navy
Washington, D. C. 20361

312

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NADC-80021-60	2. GOVT ACCESSION NO. AD-A094 679	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) NATURAL EXPOSURE OF SELECTED GRAPHITE/EPOXY COMPOSITE MATERIAL SYSTEMS		5. TYPE OF REPORT & PERIOD COVERED FINAL REPORT 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) R. E. TRABOCCO		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Aircraft and Crew Systems Technology Directorate NAVAL AIR DEVELOPMENT CENTER (60) Warminster, Pennsylvania 18974		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS AIRTASK 62761N ZF61-542-001 Work Unit ZM501
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Air Systems Command Department of the Navy Washington, D. C. 20361		12. REPORT DATE 31 July 1980 13. NUMBER OF PAGES 66
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Mechanical Properties
Environmental Degradation
Composites
Graphite/Epoxy

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A study was made on selected graphite/epoxy composite materials exposed at different geographical locations. The Panama exposure site was more detrimental to reduced static strength than the Warminster, PA site. Coating was found to negate irreversible ultraviolet plus moisture degradation. Reversible elevated temperature moisture degradation was approximately 25% for matrix dominated properties. The T300/5208 system offered the best resistance to degradation from natural exposure. The use of a 6 ply $0^{\circ} \pm 45^{\circ}$ panel loaded in sustained flexure was found to enhance scatter.

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SUMMARY

INTRODUCTION

Increased utilization of graphite/epoxy (GR/E) composites in Naval aircraft has placed increased emphasis on reliable performance of these materials. Undoubtedly, these materials offer significant advantages over conventional monolithic metals. There are specific areas of concern, however, regarding polymeric materials and composites and one of the most important is ascertaining the extent of environmental degradation.

This Center is currently involved in a study to determine the influence of various environments on emerging graphite/organic matrix composites. This work is funded by the Naval Air Systems Command and is carried out under AIRTASK 62761N ZF61-542-001, Work Unit ZM501 titled, "Data Base for Graphite/Organic Composites." This report covers the results of the initial phases of this program and is principally concerned with natural weathering of seven graphite/epoxy composite materials.

SUMMARY OF RESULTS

Natural exposures were conducted on seven graphite/epoxy composite laminates at sites in the Canal Zone, Panama and Warminster (Naval Air Development Center), Pennsylvania. These materials were Modulite 5206 (100 Series), T400 / 2544 (200 Series), 3002/T (300 Series), Modulite 5208 (400 Series), AS/3501 (500 Series), Fortafil 4R/SP286 (600 Series) and T300/520B (700 Series). All laminates were 6" x 9" panels in a 6 ply $0^o \pm 45^o$ orientation prestressed in a static deflection flexural mode. Equal numbers of panels were exposed coated and uncoated. Periodic retrievals were conducted at 1, 3, 6, 12, 18, 24, 30 and 36 months. Weight measurements were performed on a before and after basis as were static tensile, compression and shear tests at room temperature. In specific cases mechanical properties were also conducted at elevated temperature.

The uncoated 100 - 700 Series materials evidenced varying degrees of irreversible degradation due to moisture and ultraviolet exposure. This deterioration manifested itself in resin loss, loose fibers, and some matrix cracking mostly at the exposed panel surfaces. This type of degradation was measurable by loss in 0^o outer ply load carrying capability such as 0^o tension or 0^o compression data. The 200 Series material evidenced the worst case of this type of deterioration with permanent warping and material loss while the 700 Series was the most resistant to this particular form of degradation. This type of degradation was initiated after approximately 18 months exposure in Panama.

Coating seemed to negate to a great extent the irreversible degradation due to moisture and ultraviolet irradiation. Coating removal during exposure was manifested to some degree and was more apparent with Panama exposures than for those panels exposed at Warminster.

Reversible degradation, that observed at elevated temperature on matrix dominant properties and due to moisture, was encountered on coated as well as

uncoated specimens. Degradation approximating 25% in matrix dominant properties was observed in the 700 Series material when tested at 250° F.

Weight measurements taken on exposed panels indicated higher moisture contents with Panama exposures than Warminster exposures. There was only a slight weight change with seasonal change for panels exposed at either site. Painted panels were found to absorb more moisture than unpainted panels. Saturation levels were typical of graphite/epoxy 350° F curing systems with the exception of the 200 Series material which was somewhat higher. Some unpainted panels tended to offset weight increases due to moisture absorption with resin loss with increasing exposure time.

Post aging was in evidence for some of the exposed panels. This reflects processing and or chemical constituent deficiencies for the respective materials. This and the use of a prestressed 6 ply 0° ± 45° laminate were felt to contribute to the greater than anticipated scatter in residual static strength.

CONCLUSIONS

1. All exposed uncoated composite materials underwent some degree of irreversible degradation which varied with the particular composite system. The 700 Series material was found to be the most resistant to this type of degradation while the 200 Series material behaved the poorest in this regard. This deterioration was shown by loss in room temperature static mechanical properties and was accompanied by resin loss from the exposed surface.

2. Coating, to a great extent, eliminated surface type irreversible degradation, but there was no significant difference between coated and uncoated panels regarding reversible shear moisture degradation.

3. Moisture levels of coated panels were typical of saturation contents measured in laboratory studies with the Panama exposures evidencing higher levels of saturation than the Warminster panels.

4. Panama exposures evidenced greater degrees of deterioration than those panels exposed in Warminster.

5. The use of a prestressed 0° ± 45° 6 ply panel tended to accentuate environmental degradation.

6. Scatter in laminate residual mechanical property strength was aggravated by processing and chemical characterization deficiencies and by the use of a 6 ply 0° ± 45° laminate.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

BACKGROUND

An important consideration with respect to the use of advanced polymeric composites in Naval aircraft is the effect of exposure of these materials to he service environment. It has become clear in recent years, (reference (a), (b), and (c)) that properties of composite materials are greatly affected by environmental exposure. One result is that it is not possible, at present, to take full advantage of the attractive properties of these materials until the changes which occur on exposure are more fully documented and understood.

A principal environmental contaminant is moisture which is absorbed by the composite polymer matrix material and which diffuses, given time, throughout the entire composite structure. It is generally thought that moisture causes degradation of matrix dominated mechanical properties by plasticizing the resin and thus lowering its glass transition temperature (Tg). The polymer structure has an affinity for water through highly polar molecular groups in the cured resin. This affinity causes a disruption of secondary bonds between adjacent polymer chains as opposed to a direct irreversible cleavage of primary bonds. As a consequence, the resin dependent strength properties of moisture laden Graphite/Epoxy Laminates deteriorate significantly during thermal exposures above the reduced glass transition temperature. The extent of the deterioration is dependent on the amount of absorbed moisture and the test temperature. It has been shown that the phenomenon is reversible and will vanish on baking out the specimen for extended times at an elevated temperature, under vacuum. The moisture effect manifests itself with the matrix dependent mechanical properties only at elevated temperatures. This is in contrast to the exposure of glass/epoxy (Glass/Epoxy) where it has been shown that moisture can also irreversibly affect the glass fiber/resin interface leading to reductions in specific properties at ambient temperature, reference (d).

Moisture conditioning applied in concert with thermal cycling close to the glass transition temperature of the matrix material can irreversibly affect the mechanical and physical properties if the thermal cycle peak exceeds the glass transition temperature of the material. In this case micro cracking of the matrix can occur (reference (e)) and higher than equilibrium concentrations of moisture will be absorbed by the composite with resulting increases in diffusivity and further decreases in specific mechanical properties. The diffusivity is increased because the lower Tg resin is a softer medium for water to diffuse through and can more easily deform or undergo viscuous flow to accommodate the water.

Natural exposures of composite laminates show that some fluctuations occur in moisture content with seasonal changes, reference (f). The extent of these weight changes will be affected by laminate thickness, exposed surface area, edge considerations as well as other factors. Practical considerations of moisture diffusion in thick laminates point to years to achieve equilibrium.

With natural exposure there are other environmental variables which can influence the mechanical behavior of Graphite/Epoxy composites. Ultraviolet radiation can cause chain scission in the epoxy resins. Normally, ultraviolet light or high energy radiation is required to break the covalent bonds in the polymeric matrix. Irradiation with ultraviolet light also leads to photooxidation proceeding through radical reactions. These reactions are similar to thermal-oxidation, with light serving to produce radical sites. The radicals generated can then react further with oxygen to form peroxide radicals, which in turn react to yield other products. Temperature fluctuation, ozone, and ultraviolet radiation in concert with moisture absorption all take their toll on polymeric materials depending on the type of material, location and length of exposure.

This report describes the result of a continuing investigation into real time exposure effects on specific organic matrix composite systems. Three geographical locations were selected as the exposure sites for the Graphite/Epoxy systems studied. These were Coco Solo, Panama and Warminster, Pennsylvania and, to a much lesser extent Kure Beach, North Carolina. The site locations were chosen to provide a variation in climatic conditions and to note the accompanying changes in composite properties with time.

The Warminster, Pennsylvania exposure site is typical of the four season variation of the northeastern United States. However, the exposure site in the Canal Zone represents more extreme environmental conditions. Table I shows some typical environmental data for the various regions of the Canal Zone. It should be noted that the exposure site is on the Atlantic side of the Canal Zone where the humidity conditions are highest. Typical diurnal variations of temperature and himidity are shown in Figures 1 and 2 for this portion of the Canal Zone. Figure 3 shows the monthly variation in solar radiation for the various regions of the Canal Zone. For comparison of ultraviolet or the short wavelength portion of the solar radiation spectrum, Yuma, Arizona typically has 10,000 langleys per year, while the Canal Zone might evidence 8,500 langleys per year. A surface exposed in Panama would see approximately 28 langleys in the dry season and 20 langleys for a similar exposure during the wet season (daily averages). Correspondingly, similar exposures in Yuma, Arizona would show a greater change specifically, 28 langleys in summer and 14 langleys in winter. So not only is the ultraviolet radiation relatively high in the Canal Zone but it does not change greatly from the wet to dry season.

EXPERIMENTAL PROCEDURE

The materials investigated in this study were all graphite/epoxy (Gr/E) matrix composites. This effort had its genesis in December of 1972 when the 100 - 500 Series materials were exposed at Panama and Warminster, Pennsylvania. Six months later the 600 Series was added and in the fallof 1974 the 700 Series material was incorporated into the program. The designations used for these various materials are shown in Table II.

All materials were supplied in prepreg form from which panels were fabricated and exposed at the previously mentioned sites. The panels each consisted of 6 plies in a $0^{o} \pm 45$ orientation with the zero's being fabricated on the outside of the laminate. Half of the panels were coated with an epoxy polyamide primer (MIL-C-23377C) followed by a polyurethane topcoat (MIL-C-81773B). The panels were subjected to a sustained flexural load amounting to approximately 20% of the flexural failure load. The anodized aluminum holders were electrically insulated from the graphite/epoxy utilizing Tedlar strips at the contact areas.

Retrievals were performed periodically at the various exposure sites; Warminster and Panama exposures were retrieved at 1, 3, 6, 12, 18, 24, 30 and 36 month intervals. A limited number of specimens were exposed at Kure Beach, North Carolina and were retrieved at yearly intervals of 1, 2 and 3 years.

All testing for the 100 - 600 Series materials was conducted at room temperature. The inception of this effort was prior to the studies indicating high temperature moisture degradation in composite materials. The 700 Series data includes elevated temperature tests. Mechanical properties tests were divided into tension, compression and short beam shear determinations. The six by nine inch panels were divided so as to make 4 tensile tests, 3 compression tests and 5 short beam shear tests. On the unpainted panels, two of the four tensile specimens had biaxial strain gages affixed to them, to determine Poisson's ratio Young's modulus, strain to failure, and energy density. The 700 Series included flexural tests and a limited amount of 90° tensile data.

The tensile specimens were 1" x 9" with bonded glass tabs; the compression test configuration is depicted in the photograph of Figure 4. In this type of compression test, fingers are used to provide lateral support to the 1-1/2" x 1" specimen to prevent stability problems. In addition to the mechanical property determinations, weight measurements were made on each panel before and after exposure to determine the percent increase in moisture content; all panels were weighed immediately after fabrication and as soon as possible after exposure.

Visual examinations were performed on all exposed panels. Microexamination and Scanning Electron Microscopy (SEM) were performed on specific samples.

All data were routinely examined for significant trends. Standard deviations and coefficients of variations were determined for specimen replicates.

RESULTS

Some systems exhibited more of a dependence on exposure site than others. Figures 5 and 6 show the changes in tensile strength of uncoated 200 Series material with time at Panama and Warminster, Pennsylvania. The Warminster exposures did not exhibit as much reduced strength as those exposed at Panama.

TENSILE STRENGTH

Figure 7 shows the tensile strength of coated and uncoated 400 Series specimens as affected by exposure at Warminster. The curves show that strength is essentially unchanged with exposure time.

Uncoated specimens were strain gaged in order to obtain modulus, strain and Poisson's ratio data. Figure 8 shows the change in percent retained ultimate strain with increasing exposure time of uncoated 200 Series material exposed at Warminster, Pennsylvania. Note the initial rise then decrease in longitudinal strain with time. Figure 9 shows the same type data for uncoated material exposed at the Canal Zone site. The initial rise is greater and the following decrease in strain proceeds at a faster rate than that of Figure 8. The uncoated 200 Series material is again depicted in Figure 10 which shows the change in Poisson's ratio as a function of Panama exposure time. This particular parameter increases slowly at first and then at a markedly increased rate with longer time.

COMPRESSIVE STRENGTH

Figures 11 - 13 show percent retained compressive strength for specific uncoated Warminster exposures. These compression tests were conducted at room temperature on all 100 - 70 Series material retrievals. Figure 11 depicts the change in percent retained compressive strength with time of uncoated Warminster exposures of the 300 Series material. There is apparently little change with time except at the 36 month exposure point. Figure 12 shows similar data for the 400 Series material. Although scatter is high there appears to be an initial increase and then an apparent downward trend with increasing exposure time. Figure 13 shows the room temperature compressive strength of uncoated 700 Series material as being relatively unaffected after 36 months exposure in Warminster.

Examples of compressive strength changes with time are shown for coated Warminster exposures in Figures 14 and 15. Figure 14 evidences a rise and then gradual fall in compressive properties returning almost to the initial unexposed strength. The 500 Series material shown in Figure 15 shows an undulating type of behavior, first rising then falling and showing a tendency to repeat that behavior.

Figures 16 through 18 depict the variation of compressive strength of uncoated materials exposed at the Canal Zone site for three years. The 200 Series material behavior is shown in Figure 16. The strength increases then decreases, dropping off considerably after three years of exposure. The 500 Series material (Figure 17) exhibits somewhat similar behavior except that the rise is somewhat greater and the decrease in strength not as great. The 400 Series material is shown in the same type of plot in Figure 18.

As mentioned previously, elevated as well as ambient temperature static strength tests were conducted on the 700 Series material. The next three figures show the effect of exposure on 250° F compressive strength for the 700 Series material. Figure 19 shows unpainted Warminster exposures evidencing a gradual decrease in strength. Figure 20 shows a similar plot for painted Panama exposures and Figure 21 shows the unpainted Panama exposure's compressive strength.

SHORT BEAM SHEAR STRENGTH

Short beam shear tests were conducted on all exposed panels. The 100 - 600 Series material shear tests were all conducted at ambient temperature, while the 700 Series tests were conducted at ambient and elevated temperatures. Figures 22 and 23 show the variation of shear strength for uncoated Warminster exposures of the 400 Series and 500 Series materials, respectively. Figures 24 and 25 show the interlaminar shear strength for 100 Series material exposed at the Warminster site in the coated and uncoated condition, respectively. Figure 26 shows a plot of shear strength versus time for the uncoated 300 Series material exposed at Panama. Figure 27 represents 250° F interlaminar shear strength of the uncoated 700 Series material exposed at Panama.

Flexural testing was added to the mechanical property determinations for the various exposures beginning with the 700 Series material. Flexural tests were conducted at ambient and elevated temperature conditions. Figure 28 shows the 250° F flexure strength of the uncoated 700 Series material as a function of exposure time at the Canal Zone site. A similar plot is depicted in Figure 29 for coated 700 Series material.

The static residual properties of the 600 Series were consistently lower than any other material even with unexposed panels. Because of this the 600 Series data was excluded from presentation in the report.

MACRO AND MICRO EXAMINATION

Sections of selected tensile specimens removed from panels have been chosen to show some of the effects of material exposure. Figures 30 and 31 show macrographs of sections of uncoated Panama exposures and compares them with similar unexposed materials. The tensile fractures are still evident in some of the specimens depicted in the photomacrographs. The significance of these photomacrographs is the change in surface appearance from control to exposed panels. Specifically, uncoated specimens tend to reveal an obliteration of the original "peel ply" imprint evident on control specimens along with loose fibers evident on the exposed panel surface. For coated specimens, changes in contrast, peeling or pitting should be looked for in the topcoat of exposed panels. Figures 32 and 33 are representative of coated Panama exposures while Figures 34 and 35 are indicative of control and exposed coated and uncoated Warminster exposures.

In order to obtain a thorough analysis of the state of deterioration for various samples, sections were removed, mounted, polished, and examined. Cross sections of specific samples are shown in Figures 36 through 40. The representative exposed sections are shown with their unexposed counterparts. The effects of exposure on uncoated specimens were manifested in removal or thinning of the exposed 0° ply with cracking and or delamination. Any deterioration of coated sections would be evident in a much more subtle fashion principally through coating thinning.

All panels were weighed prior to exposure and again on retrieval. Figure 41 shows the percent weight change for selected coated panels exposed at Warminster. The data for Panama exposures was similar with slightly higher rates of weight increase and levels of saturation.

LAMINATE ORIENTATION ANALYSIS

It is important to consider the role of panel orientation on static property results. In many circumstances panel configuration can have a profound influence on properties. Failure criteria for a given configuration, therefore, must be established in order to determine the significance of orientation as it affects the properties determined. In order to determine such criteria, it is necessary to have the specific material properties for the different composite systems. Table III lists the properties necessary to determine the stress distribution through the various plies. These data were obtained from the various manufacturers of graphite/epoxy composite materials. These particular properties are only listed for some of the materials included in the investigation. Parameters of interest were modulus, E and G, Poisson's ratio, ν , ultimate tensile strength, F^t, ultimate compressive strength, F^c, and elongation to failure (tension, ϵ^t, and compression, ϵ^c). Most of the properties are given in two directions. Stress distribution as a function of ply orientation shown in Figure 42 was arrived at using the values in Table III and laminated plate theory, reference (g). The effect of thermal stress due to cure cycle was superimposed on the effect of applied load. The thermal stress analysis was accomplished using the formulation of Tsai, reference (h). Figure 43 depicts the resultant stresses on the laminate used in this study.

By using a comparison of maximum strain, it is possible to determine which ply or plies control failure under a given external load. Table IV depicts the ratios of strain on a particular ply in a particular direction to the maximum strain for the laminate.

Coefficient of variation data were generated for the various exposures and are shown in Table V as averages for specific static properties over the three year exposure period.

Comparative degradation trends were established for specific materials and are shown in Figures 44 through 47.

D I S C U S S I O N

It is interesting to note the changes in properties of the various materials with increasing exposure time. Exposure site has to be considered in the context of its role in enhancing panel degradation. A material which exhibits different behavior, be it exceptional or deficient, should be noted and examined closely in order to establish the basis of such a response.

200 SERIES

The 200 Series material showed significant changes in uncoated tensile properties with increasing exposure time (Figures 5 and 6). This material exhibited a greater decrease in tensile strength than any of the other exposed materials.

It is clear from Figures 8 - 10 that the 200 Series show a diminished strain capacity in the 0^o direction. The degradation to the 0^o plies also results in increases in Poisson's ratio.

The variation of uncoated compressive strength with time also followed a similar pattern (Figure 16). Both properties showed an initial increase then a decrease in strength with continuing exposure. This phenomenon is undoubtedly due to post aging of the material on exposure.

Visual examination of this series after exposure showed permanent warping after approximately 18 months of exposure in Panama in the uncoated condition. Substantial resin loss on exposure is apparent (Figure 30); since the peel ply imprint on the unexposed panel in the macrograph has been obliterated after 36 months exposure in Panama and is replaced by evidence of loosely adherent fibers at the surface. This material also exhibited poor adhesion of the coating system with increasing exposure time in comparison to the other exposed series (Figure 33). Photomicrographs of uncoated 200 Series material disclosed cracking and an "erosive" type attack on the exposed panel surface (Figure 36B). Also evident from Figure 40 and characteristic of this test series are resin rich islands that completely separate portions of the 0^{o} ply. Resin degradation in these islands would serve as crack initiation sites which could propagate across the surface or into the interior of the material. Such a microstructure would make this material less resistant, in the uncoated condition, to the combined effects of ultraviolet radiation and moisture as compared to that of the 100 Series material shown in Figure 36A. Coating seemed to negate this type of cracking and erosive type attack, with no cracking even after 3 years of exposure (Figure 38B).

The 200 Series material contained the highest equilibrium concentrations of moisture of all the exposed materials. Undoubtedly the large resin islands and laminate cracking contributed to this observed phenomenon.

The general behavior of the other materials regarding moisture absorption was similar to the 200 Series specimens. It is apparent from Figure 41 that saturation does not occur with any of the exposed materials until about 9 months after exposure. This would coincide with the environmental conditions at Warminster. The panels were exposed at the beginning of December in 1972, subsequently, ambient humidity would peak the following summer, 8 - 9 months later. So the weight measurements for painted panels bear out the increased humidity of the warmer months in the Northeastern United States. The larger surface area to edge area ratio of these panels precludes any rapid changes in weight with exposure since surface moisture diffusion is slower than edge diffusion.

It is interesting to note that 0^{o} tensile strength, a fiber dominant failure mode which is usually not an indicator of environmental degradation, in the 200 Series material does show a significant decrease. One of the principal reasons for this is the distribution of large resin islands in the 0^{o} plies.

This particular material was most severely degraded in the uncoated condition. Exposure at the Panama site resulted in more degradation for this material than the Warminster location, however, even the latter case resulted in appreciable degradation. It appears that initial degradation for the 200 Series material exposed in Panama is detectable after 6 months exposure while the Warminster exposures do not show significant changes until 6 - 12 months later.

300 AND 600 SERIES

The 300 and 600 Series materials were similar in their resistance to degradation. Both evidenced marked effects of erosive type behavior in the uncoated condition. The moisture absorption characteristics of the 300 Series material showed this system absorbed more moisture than the others except for the 200 Series material.

100 SERIES

The overall response of the 100 Series material to three years of natural exposure was somewhat better than the 300 or 600 Series systems. Again this material in the uncoated condition showed erosive type surface degradation (note loss of surface material in Figure 36A). There was no significant effect on tensile properties as was the case with the 200 Series material. Compressive properties, however, were affected by environmental exposure.

Shear properties as measured at room temperature (Figures 24 and 25) showed no significant changes after natural exposure for three years. The maximum interlaminar shear stress occurs at the midpoint of the thickness, a region least sensitive to the effects of ultraviolet and moisture. This phenomenon was similar with all the materials exposed and tested.

400 AND 500 SERIES

The 400 and 500 Series material exhibited better resistance to natural weathering than the 100 Series system. Both, however, exhibited reductions in uncoated room temperature compression strength (Figures 17 and 18) particularly after exposure in Panama. Both also reflected surface type deterioration (Figures 30 and 31) with the 400 undergoing less damage than the 500 Series i.e., more loose fibers evident in the macrographs of tensile specimen sections. A comparison of 400 Series specimens in Figures 31 and 34 shows the difference in degradation as a function of exposure site. The 400 Series specimen from the Warminster site exhibited less loose fibers and resin loss than that from the Panama site. The microstructure of the 400 Series material was relatively uniform with no excessive amounts of resin globules, however, there was some loss of surface material (Figure 37A). Neither the 400 nor 500 Series showed any evidence of significant cracking in their microstructures after exposure.

Figure 15 shows an undulating type behavior in coated compression strength for the 500 Series material. This behavior was observed with other exposures. The state of chemical characterization knowledge was very limited when these materials were obtained from various vendors. Processing had not been optimized at that point since some of the materials were relatively new and the cure cycles were utilized on the basis of vendor information. There was no large scale production of components for any of the materials utilized in this study, hence no real optimization, by industry, of processing parameters.

700 SERIES

Of all the materials studied the 700 Series system provided the best all around resistance to environmental degradation. This system's ambient compressive strength showed little effect after three years of exposure (Figure 13). There was some surface deterioration but not as much as for any of the other resin systems. Figure 35a shows that the peel ply imprint (from initial fabrication) is still evident. It is interesting to note that some coating deterioration is reflected in Figure 35b where now the "peel ply" imprint is more clearly seen through the coating. Coating deterioration can also be seen in Figure 32B where "pitting" and interior surface patterns are becoming more clearly visible after three years of exposure.

The as-fabricated microstructures, Figure 39B of the 700 Series system, contained a distribution of fibers and no resin rich areas. The microstructures of this material appeared superior to all the others prior to any exposure.

The microstructures of coated samples are typical of those shown in Figures 38 and 39. Little or no effect of natural weathering is evident on these microstructures.

The 700 Series material was initially exposed at the end of 1974. Data of Hertz et al, reference (b) showed the effect of hot-wet testing on resin matrix dominant properties. Elevated temperature testing was, therefore, incorporated into the testing program for the 700 Series retrievals. Figure 27 shows the 700 Series shear strength at 250° F as it varies as a function of exposure time in the uncoated condition. This is typically a moisture phenomenon as opposed to a combined ultraviolet moisture effect and results in a reduction of approximately 25% in this property as compared to the unexposed 250° F strength. Practically all of this shear strength decrease is recouped by baking samples under vacuum prior to elevated temperature shear testing.

Elevated temperature compressive data for this material disclosed significant reductions in strength, Figures 19 - 21. Generally, the result is that the tropical exposure (Panama) is more deleterious to strength especially with uncoated panels. The effects were reversible on heating painted panels, particularly Warminster exposures. With uncoated panels some of the strength degradation was reversed by heating at elevated temperature for an extended period of time but compressive strength did not return to unexposed strength values. Typical hot (250° F) compressive strength of the uncoated Panama exposure (700 Series) was about 30% less than unexposed values.

700 Series exposures were subjected to elevated temperature flexure testing which indicated a reduction in strength of about 25% for the unpainted three years uncoated Panama exposures. The data for painted specimens indicated less of a reduction in 250° F flexural strength for 36 month exposures. The reduced strength of the uncoated material versus the coated samples indicates that some ultraviolet damage has occurred. The flexure properties are dependent on outer fiber strength. In this case the outer fibers are the main load carrying component for 0° flexure, and are damaged by sunlight and moisture exposure.

INFLUENCE OF SCATTER

One phenomenon that surfaced in this study was the significant amount of scatter (Table V) in the data. It is felt that the prestressed 6 ply (0 ± 45) laminate contributed to increased scatter. A more detailed examination of the possible failure modes for this type of laminate will help clarify this phenomenon.

A 1,000 lbs./in. unidirectional tensile load is depicted in Figure 42 on the 100 Series material. Sketch 42(A) shows that most of the tensile load is borne by the 0^o plies (approximately 75 ksi) as opposed to something much less for the 45^o plies. The nominal stress to thickness for this laminate is approximately 65 ksi and 0.030, respectively. However, because of the Poisson's ratio mismatch (between the 45^o and 0^o plies), there is an induced compressive stress on the outer plies and a corresponding tensile stress on the inner plies (Sketch 42(B)). The last Sketch, Figure 42(C) depicts the effects due to shear coupling on the laminate. Negative shear stresses are induced on the outer 45^o plies and positive shear stresses on the inner 45^o plies. The net result is that the failure mode for unidirectional tensile loading is unchanged and essentially controlled by the 0^o plies (fiber controlled failure).

The effect of heating as in a cure cycle is superimposed on the applied load and is shown in Figure 43. The effect is a linear one, and a temperature excursion in excess of 100^o F would simply be reflected in corresponding higher values of stress. From Figure 43(A) through 43(B) the results are similar to those depicted in Figure 42 and the magnitude of the stresses are not sufficient to alter the failure mode.

Taking this loading consideration of 1,000 lbs./in. it is possible to determine the strains in various directions for the different materials as shown in Table III. Further, knowing the maximum laminate strain and comparing it to the strain in a particular ply in a particular direction indicates that the greatest percentage of the total strain is borne by the 0^o plies. The ratio of strain in the 0^o plies to the total strain to failure in the composite is an order of magnitude greater than any other similar ratio.

Failure under 0^o compressive or tensile loading for this orientation is controlled by the 0^o ply. That means one prestressed 0^o ply will have a significant bearing on the mechanical properties of the exposed panels. Any surface deterioration could have a substantial effect on these surface plies. Thus, it can be seen that a prestressed laminate of this type can lead to increased scatter in residual mechanical properties especially after environmental exposure.

TRENDS IN STATIC PROPERTIES WITH NATURAL EXPOSURE

All of the previous data presented represent the actual static strength after specific exposure periods. It is meaningful to discuss the general trend of specific materials for a particular property in a comparative fashion. Specific properties of selective materials have been chosen for representation in this manner.

Figure 44 represents the trend curves of 200 and 400 Series residual tensile strength. The 400 Series trend was typical of all materials with the exception of the 200 Series. The 200 Series shows post aging behavior followed by significant degradation.

The "typical" degradation in compression strength is shown for selected materials in Figure 45. Some materials exhibit post aging while others reflect more subtle changes in strength. The 700 Series material evidences the least amount of strength change with time while the 200 Series reflects the greatest perturbations in residual compression with exposure.

Since the 700 Series material was the only one tested at elevated temperatures it cannot be compared with other systems. However, Figure 46 reflects a comparison of exposure site on elevated temperature compressive strength and Figure 47 shows coating influence on high temperature flexure strength. These comparisons demonstrate the tendency of the more severe degradation at the Panama site over that of Warminster and the benefits of coating.

CONCLUSIONS

1. All exposed uncoated composite materials underwent some degree of irreversible degradation which varied with the particular composite system. The 700 Series material was found to be the most resistant to this type of degradation while the 200 Series material behaved the poorest in this regard. This deterioration was measured by room temperature static mechanical properties and was accompanied by resin loss from the exposed surface.

2. Coating, to a great extent, eliminated surface type irreversible degradation, but there was no significant difference between coated and uncoated panels regarding reversible shear strength moisture degradation.

3. Moisture level of exposed coated panels were closely duplicated by laboratory exposure to similar humidity levels. Panels exposed in Panama exhibited higher saturation levels than those exposed at Warminster.

4. Panama exposures evidenced greater degrees of deterioration than those panels exposed in Warminster.

5. The use of a prestressed ($0^{o} \pm 45^{o}$) 6 ply panel tended to accentuate environmental degradation.

6. Scatter in laminate residual mechanical property strength was aggravated by processing and chemical characterization deficiencies and by the use of a 6 ply ($0^{o} \pm 45^{o}$) laminate.

RECOMMENDATIONS

1. Time of exposure should be increased in order to obtain more comprehensive data.

2. More than one laminate configuration should be utilized to increase useful exposure data i.e., $\pm 45^{o}$ laminate for matrix dominant effects in addition to a $0^{o} \pm 45^{o}$ 90 16 ply laminate.

3. Expose new emerging organic composite materials including Kevlar reinforced composites.

4. Develop a preliminary property history of promising fiber reinforced organic composites prior to long term exposure to avoid efforts directed at unpromising materials.

5. Future candidate materials for long term environmental exposure should be based on definitive laboratory mechanical property evaluation following optimization of processing.

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NADC-80021-60

TABLE I TYPICAL WEATHER DATA, CANAL ZONE *†

	DRY SEASON ‡			RAINY SEASON §		
	PACIFIC	MID-ISTHMUS	ATLANTIC	PACIFIC	MID-ISTHMUS	ATLANTIC
Open temperature, daytime (°F)	82 - 90	82 - 89	80 - 86	82 - 87	80 - 86	82 - 86
Open temperature, nighttime and during heavy rain (°F)	65 - 72	65 - 70	68 - 74	75 - 78	70 - 73	75 - 78
Jungle temperature, daytime (°F)	80 - 85	78 - 81	79 - 81	81 - 83	79 - 82	80 - 83
Jungle temperature, nighttime (°F)	74	74	75	76	74	75
Highest temperature ever measured (°F)	102	95	103	94	95	98
Lowest temperature ever measured (°F)	63	60	63	63	68	64
Dew point, all day (°F)	71	70	75	76	73	76
Relative humidity, average lowest daily (%) ‡‡	50	56 (estimate)	70	70	71 (estimate)	73
Duration of sunshine, daily average (hrs)	8.5	8.2	8.6	5.1	4.5	5.7
Global radiation on horizontal plane, daily average (langleys/day)	500	435	520	335	315	340
Direct solar radiation on horizontal plane, daily average (langleys/day)	328	---	330	140	---	100 - 200**
Indirect solar radiation (sky radiation) on horizontal plane, daily average (langleys/day)	172	---	190	195	---	140 - 240**
Prevailing wind direction	N	N	NNE	NW	NNW	NW or S
Mean wind speed, noon (mph)	10 - 12	---	12 - 14	5 - 7	---	6 - 8
Mean wind speed, night (mph)	6 - 8	---	12 - 14	0 - 3	---	3 - 4
Rainfall, monthly average (in)	0.5 - 1	---	1.5 - 2	9 - 14 ††	---	11 - 25 ††
Rainfall, monthly maximum (in)	7.1	---	16	31	---	45
Rainfall, 24-hour maximum (in)	4.8	---	9	5.7	17	14
Rainfall, 1-hour maximum (in)	1.3	---	4.5	3.8	---	5.7

	PACIFIC	MID-ISTHMUS	ATLANTIC
Rainfall, yearly average, (in)	80	105	130
Rainfall, yearly maximum (in)	117	---	183

* Sufficient measurements are available for the Pacific side (Fort Clayton, Chiva Chiva) and the Atlantic side (Fort Sherman, Coco Slope) but are scant for the Mid-Isthmus.

† Data were derived from measurements made by the Panama Canal Company and the US Army Atmospheric Sciences Laboratory, Canal Zone Meteorological Team.

‡ Data apply to February and March - the driest months.

§ Data apply to June through November.

** Only June and July.

†† Only November.

‡‡ Maximum RH is 95 - 100% nightly for all areas in both seasons.

TABLE II DESIGNATIONS USED TO DENOTE SPECIFIC MATERIALS STUDIED IN THIS INVESTIGATION

MATERIAL	SERIES DESIGNATION
Modulite 5206	100
Thornel 400/2544	200
Hercules 3002T	300
Modulite 5208	400
Hercules AS/3501	500
Fortafil 4R/SP286	600
T300/5208	700

TABLE III GRAPHITE-EPOXY MATERIAL PROPERTIES

MATERIAL	NARMCO MODMOR II 5206	NARMCO T300 5208	HERCULES AS 3002	HERCULES HTS 3002	HERCULES AS 3501
E_1	22×10^6	20.8×10^6	18.5×10^6	22×10^6	20.2×10^6
E_2	1.9×10^6	1.46×10^6	1.63×10^6	1.35×10^6	1.35×10^6
ν_{12}	0.38	0.31	0.25	0.21	0.30
G_{12}	0.6×10^6	0.6×10^6	0.6×10^6	0.6×10^6	0.6×10^6
F_1^T	186 KSI	234 KSI	211 KSI	194 KSI	231 KSI
F_2^T	9.7 KSI	8.5 KSI	9.0 KSI	9.7 KSI	8.2 KSI
ϵ_1^T	8.52×10^{-3}	9.76×10^{-3}	12.4×10^{-3}	8.6×10^{-3}	11.5×10^{-3}
ϵ_2^T	5.24×10^{-3}	4.70×10^{-3}	4.71×10^{-3}	7.8×10^{-3}	5.3×10^{-3}
F_1^C	223 KSI	205 KSI	171 KSI	224 KSI	—
F_2^C	33 KSI	20.6 KSI	31 KSI	29 KSI	—
ϵ_1^C	11.9×10^{-3}	10.0×10^{-3}	13.7×10^{-3}	12.5×10^{-3}	—
ϵ_2^C	—	14.0×10^{-3}	10.9×10^{-3}	21.6×10^{-3}	—

TABLE IV STRENGTH AND STIFFNESS ANALYSIS

MATERIAL	NARMCO MODMOR II 5206	NARMCO T300 5208	HERCULES HTS 3002	HERCULES AS 3002	HERCULES AS 3501
N_x	1000	1000	1000	1000	1000
ϵ_1^0	3.401×10^{-3}	3.567×10^{-3}	3.390×10^{-3}	3.912×10^{-3}	3.658×10^{-3}
$\epsilon_1^0/\epsilon_1^T$	0.399	0.366	0.394	0.315	0.318
ϵ_2^0	-2.635×10^{-4}	-2.717×10^{-4}	-2.581×10^{-4}	-2.850×10^{-4}	-2.770×10^{-4}
$\epsilon_2^0/\epsilon_2^C$	—	0.0194	0.0206	0.0261	—
ϵ_1^{45}	3.829×10^{-4}	4.249×10^{-4}	4.043×10^{-4}	5.310×10^{-4}	4.439×10^{-4}
$\epsilon_1^{45}/\epsilon_1^T$	0.045	0.0435	0.047	0.043	0.039
ϵ_2^{45}	3.829×10^{-4}	4.249×10^{-4}	4.043×10^{-4}	5.310×10^{-4}	4.439×10^{-4}
$\epsilon_2^{45}/\epsilon_2^T$	0.073	0.090	0.052	0.113	0.084
E_x	8.5×10^6	8.5×10^6	8.9×10^6	7.8×10^6	8.3×10^6
ν_{xy}	0.77	0.76	0.76	0.73	0.76
$\sigma_x^{ult.}$	75.9 KSI	82.9 KSI	76.9 KSI	96.2 KSI	95.3 KSI

TABLE V

COEFFICIENT OF VARIATION AVERAGES FOR PARTICULAR STATIC PROPERTIES FOR 3 YEAR EXPOSURES

	Uncoated Warminster			Uncoated Panama			Coated Warminster			Coated Panama		
Material	C.S.	H.S.	T.S.	C.S.	H.S.	T.S.	C.S.	H.S.	T.S.	C.S.	H.S.	T.S.
100	7.2	12.7	9.0	10.9	13.9	7.2	15.8	9.7	9.9	16.4	14.1	12.0
200	10.9	14.3	9.1	19.2	9.4	8.7	13.2	11.6	10.1	11.5	15.9	7.8
300	10.6	15.2	10.6	10.7	15.1	11.9	13.1	9.6	16.2	13.1	17.1	10.5
400	6.0	13.0	13.8	10.5	10.4	18.6	20.8	14.8	14.2	15.4	9.0	14.1
500	7.9	12.8	14.6	11.3	11.4	12.4	13.9	11.8	12.7	13.4	12.9	19.6
600	15.3	7.3	8.4	11.8	10.6	10.2	11.4	9.3	8.9	14.8	9.0	15.7
			F.S.			F.S.			F.S.			F.S.
700	14.8	12.6	13.4	14.3	9.4	10.0	9.6	11.6	9.3	8.0	12.0	12.4

C.S. Compressive Strength
H.S. Horizontal Shear Strength
T.S. Tensile Strength
S.S. Flexural Strength

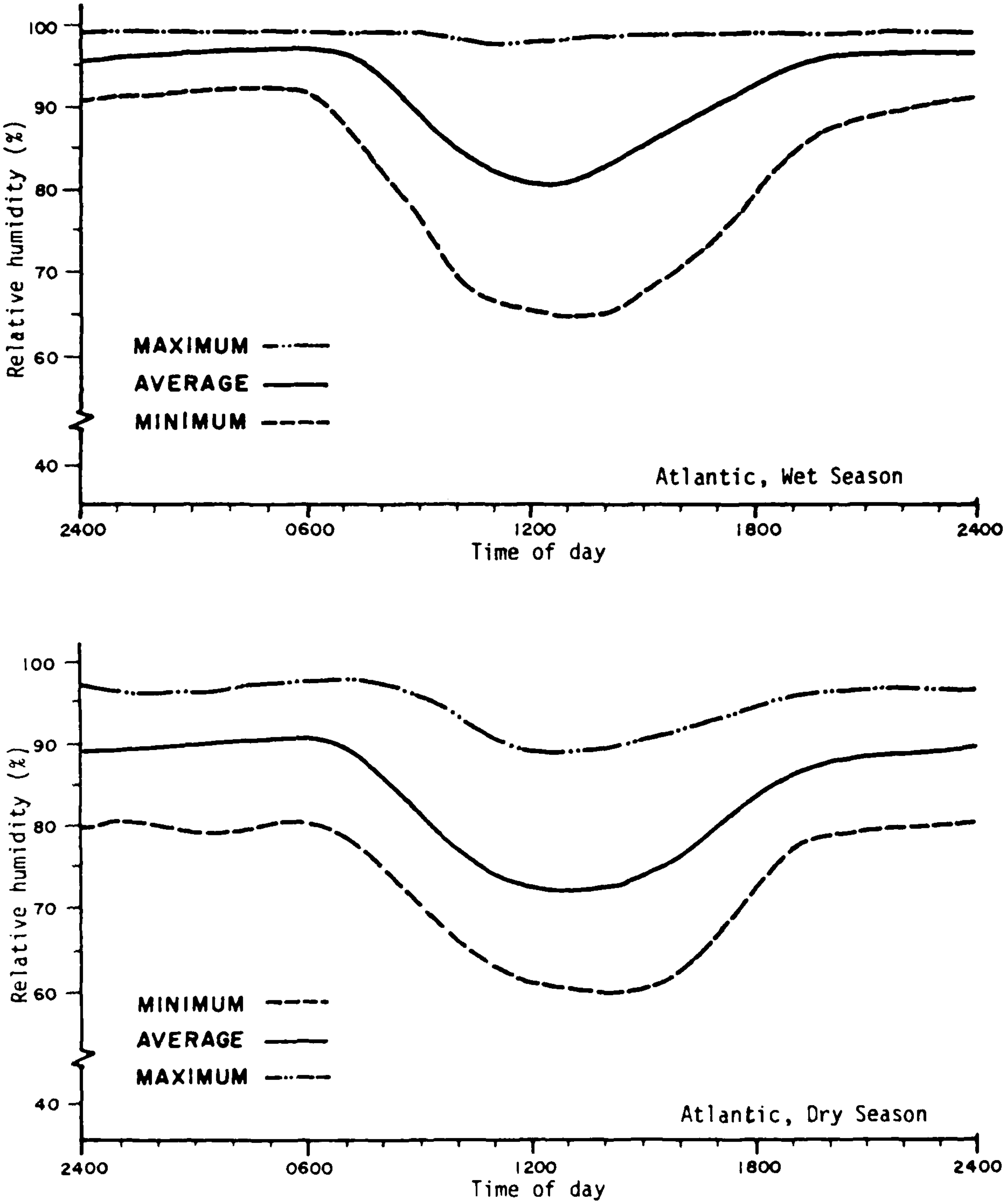


FIGURE 1 RELATIVE HUMIDITY - ATLANTIC AREA OF CANAL ZONE

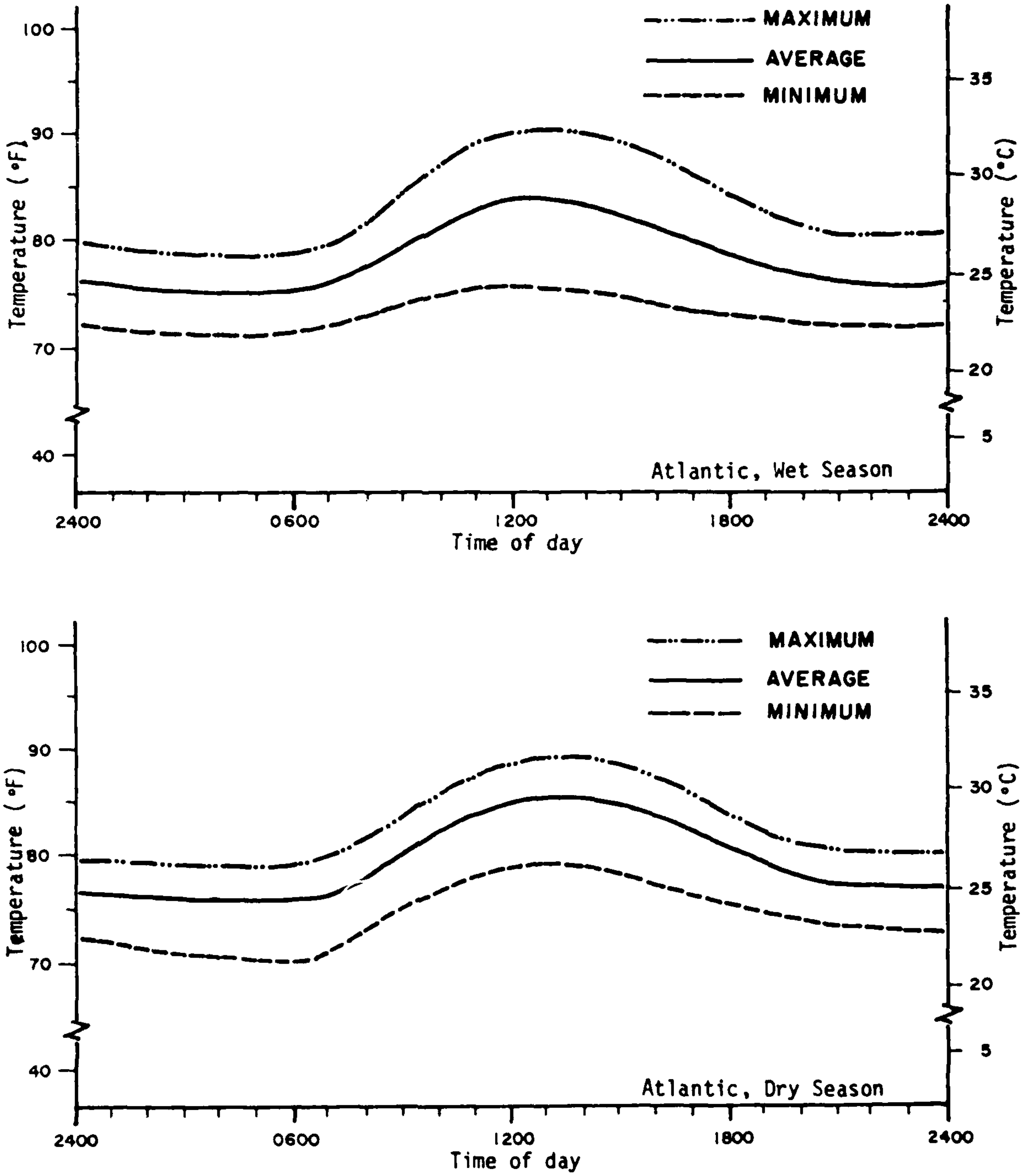


FIGURE 2 ATLANTIC DIURNAL TEMPERATURE PROFILES OF THE CANAL ZONE

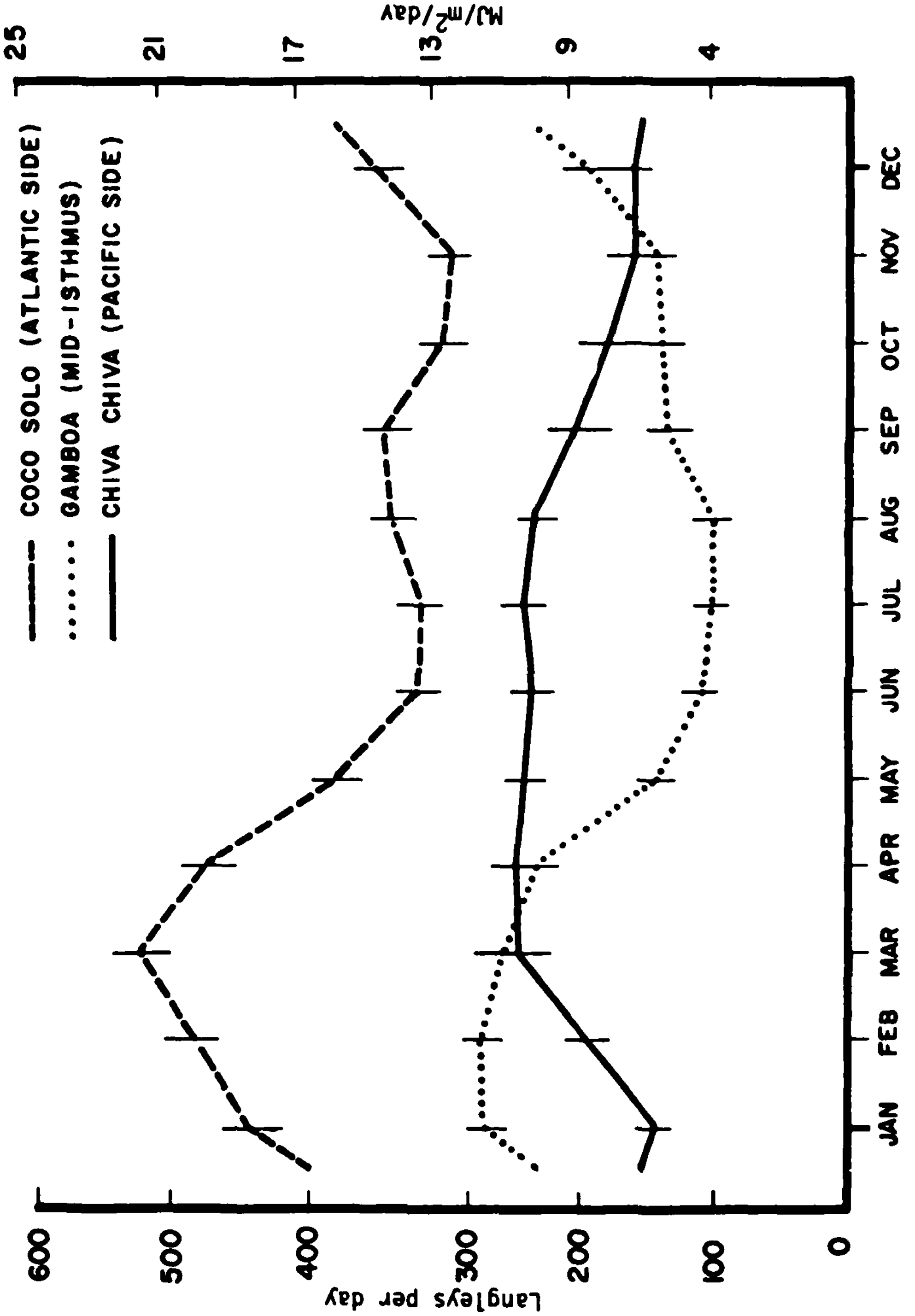


FIGURE 3 MONTHLY VARIATION OF THE SOLAR RADIATION - ATLANTIC SIDE OF THE CANAL ZONE

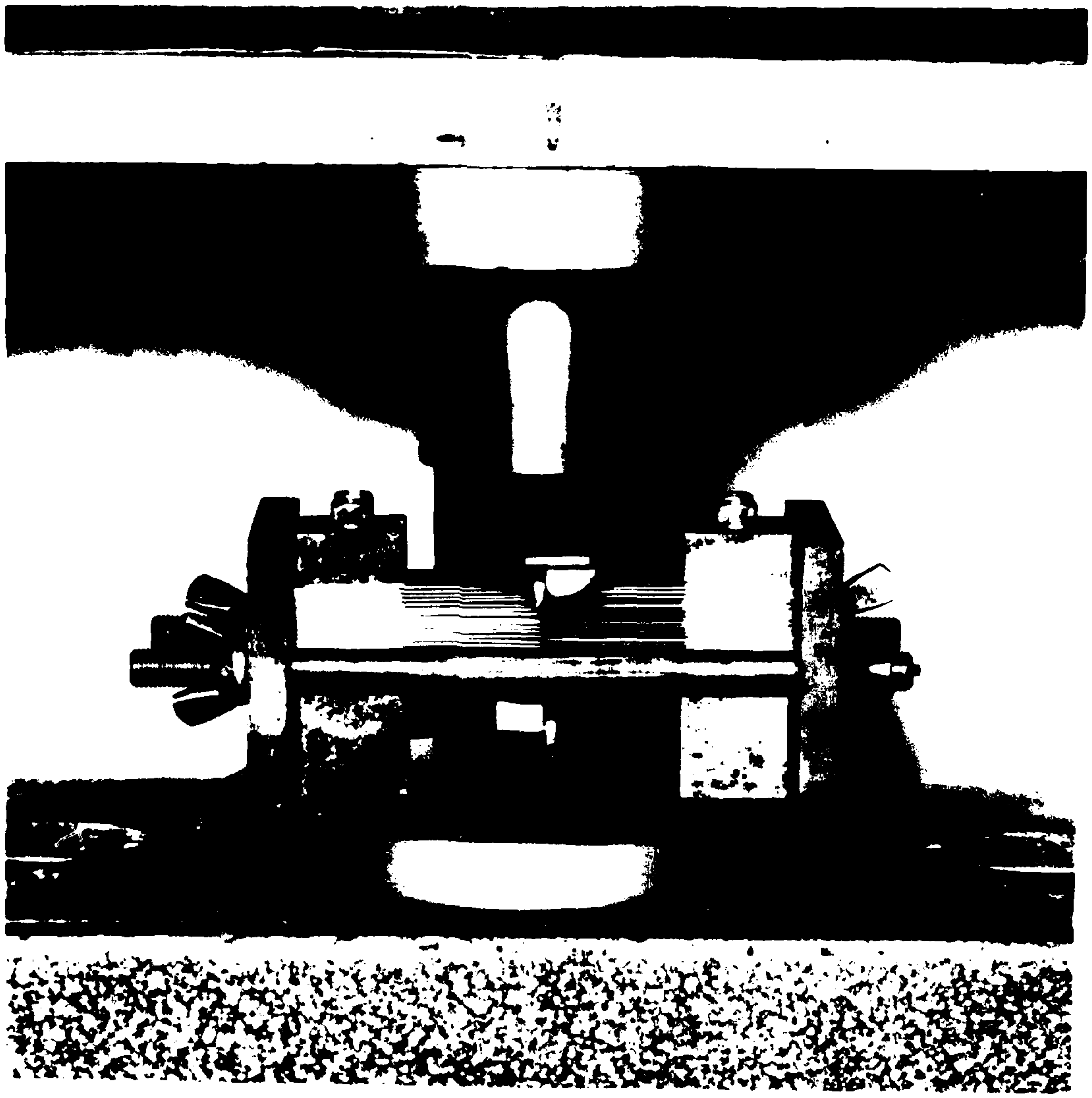


FIGURE 4 PHOTOGRAPH OF THE COMPRESSIVE TEST FIXTURE USED IN THIS STUDY (½x)

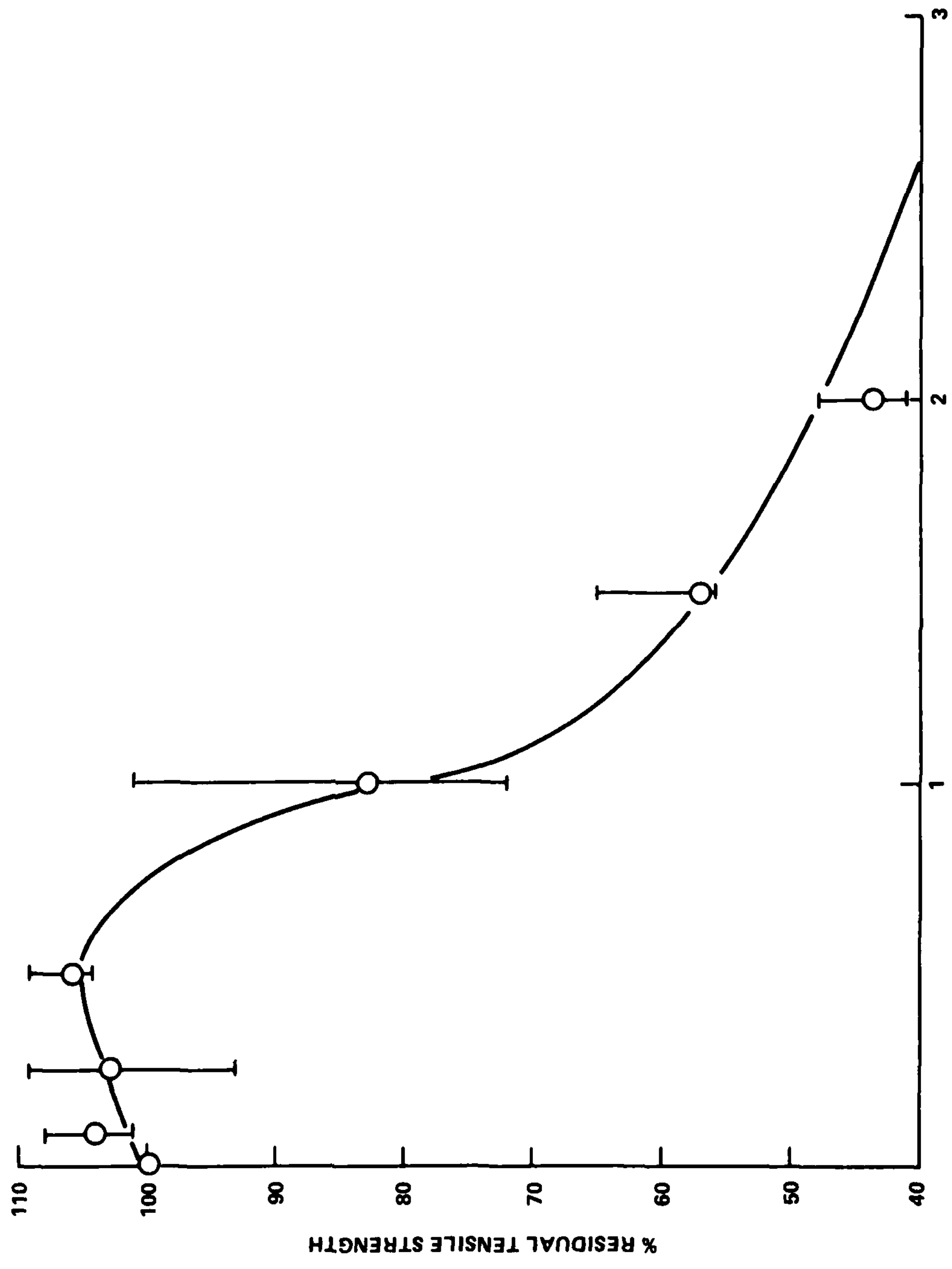


FIGURE 5 THE VARIATION IN RESIDUAL TENSILE STRENGTH VERSUS EXPOSURE TIME IN PANAMA (200 SERIES UNCOATED)

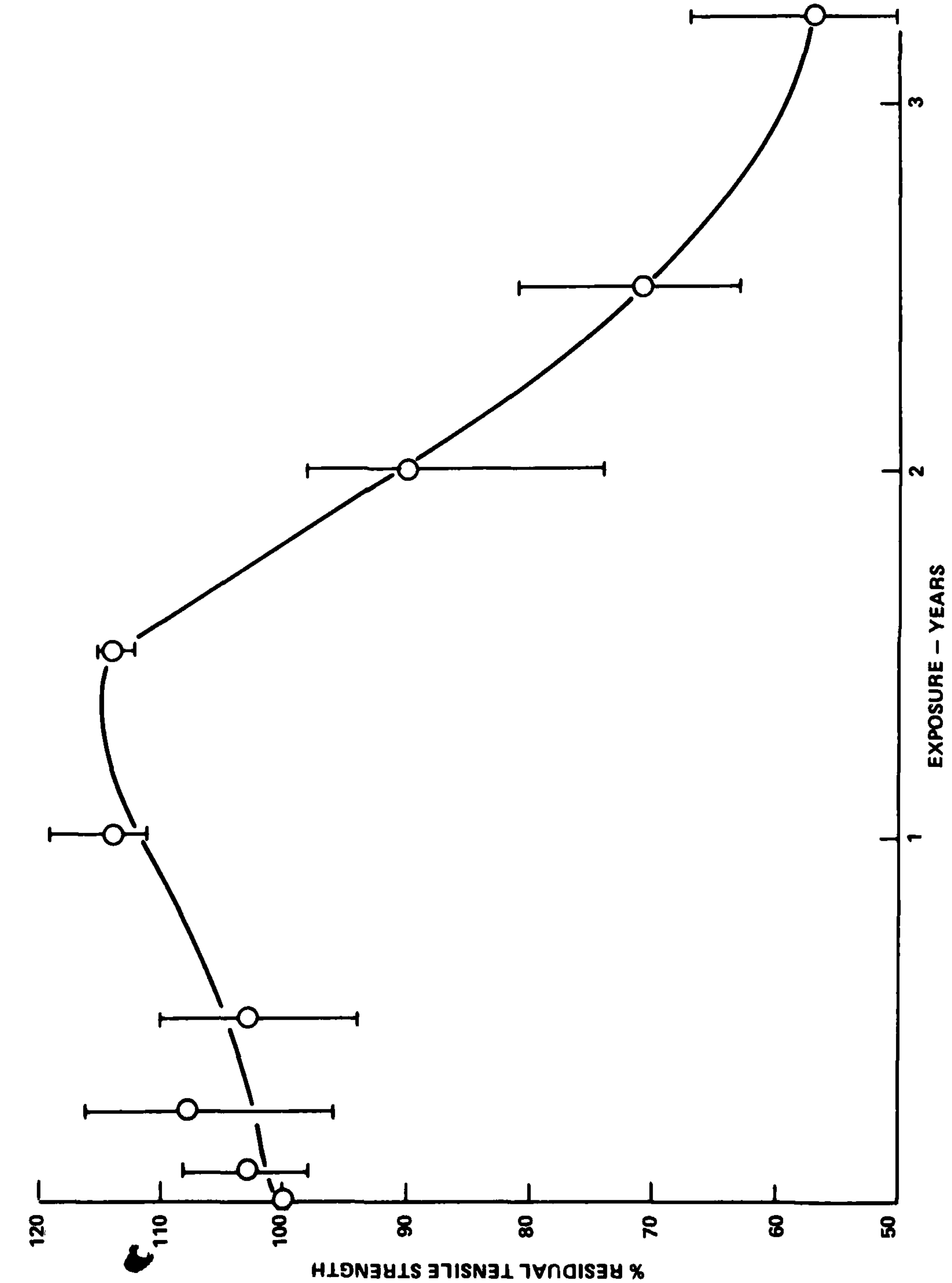


FIGURE 6 THE VARIATION IN RESIDUAL TENSILE STRENGTH VERSUS EXPOSURE TIME IN WARMINSTER (200 SERIES UNCOATED)

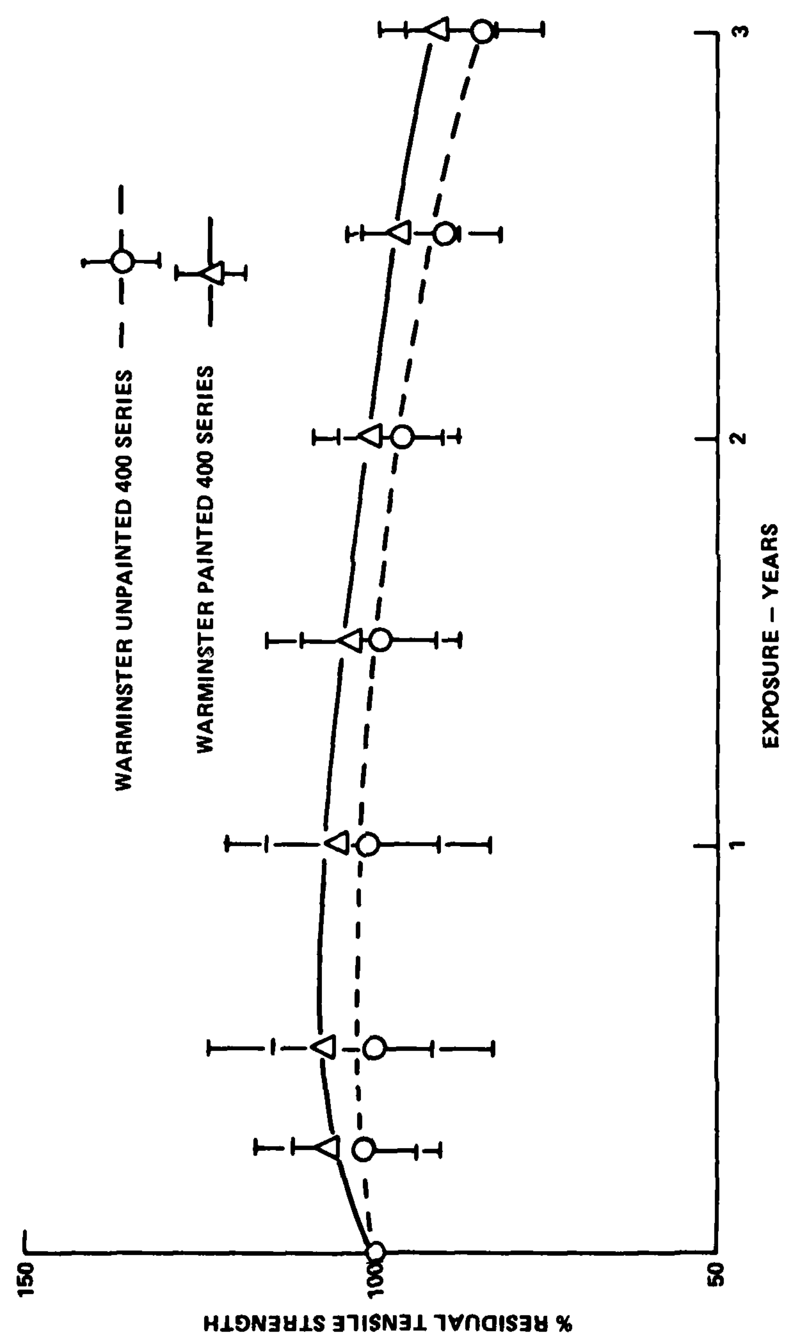


FIGURE 7 THE VARIATION IN RESIDUAL TENSILE STRENGTH VERSUS NATURAL EXPOSURE TIME IN WARMINSTER

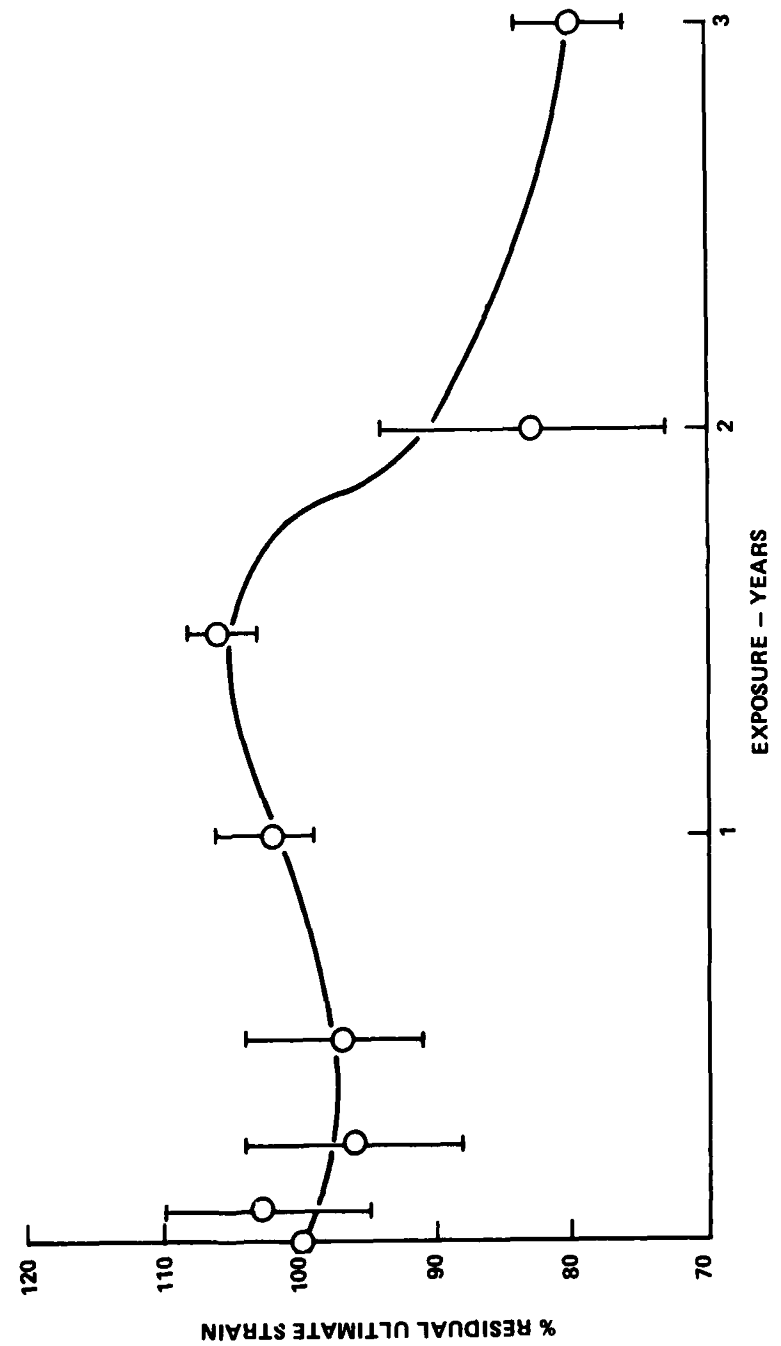


FIGURE 8 RESIDUAL TENSILE STRAIN (0^o) VERSUS EXPOSURE TIME IN WARMINSTER (200 SERIES UNCOATED)

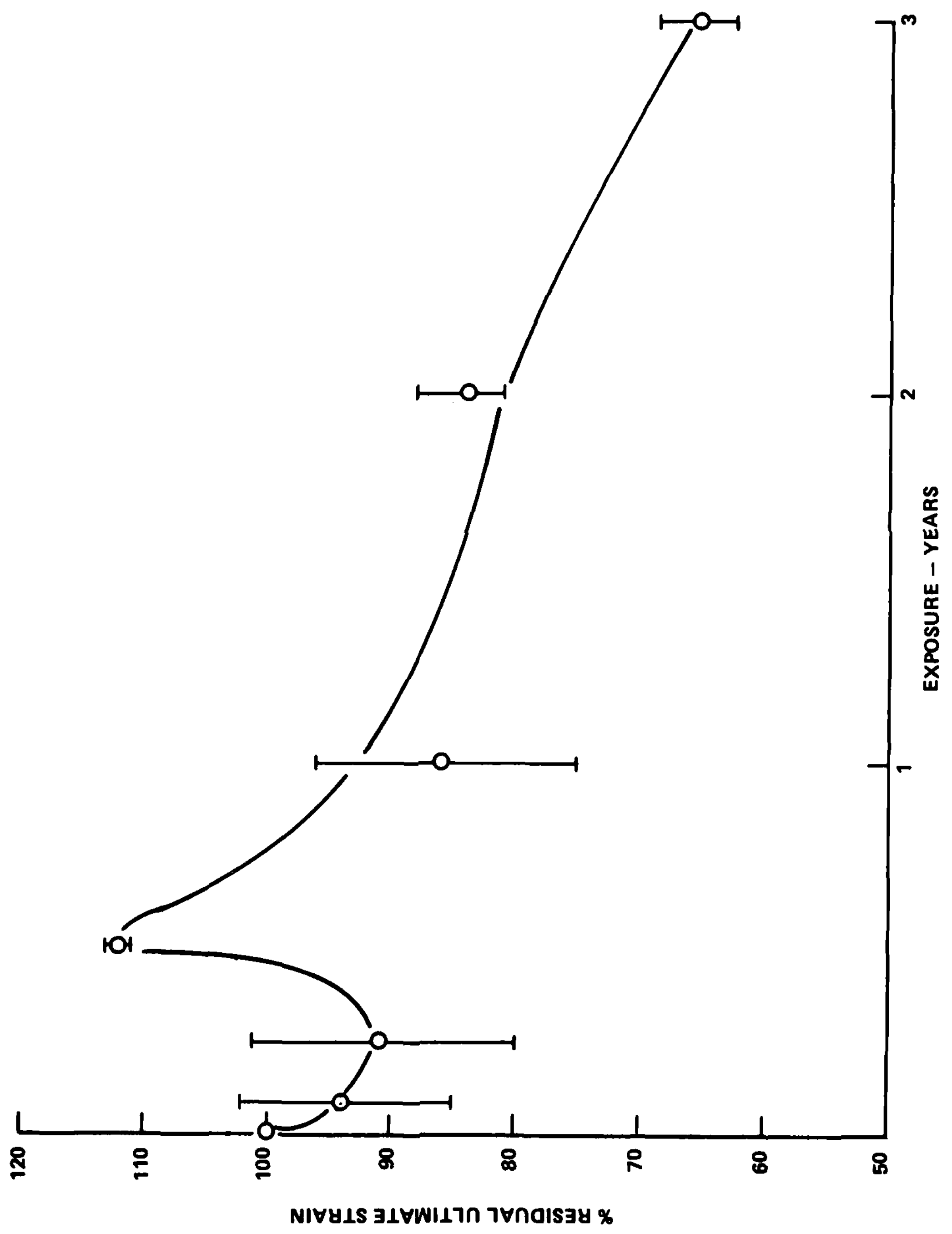


FIGURE 9 RESIDUAL ULTIMATE STRAIN VERSUS EXPOSURE TIME IN PANAMA (200 SERIES UNCOATED)

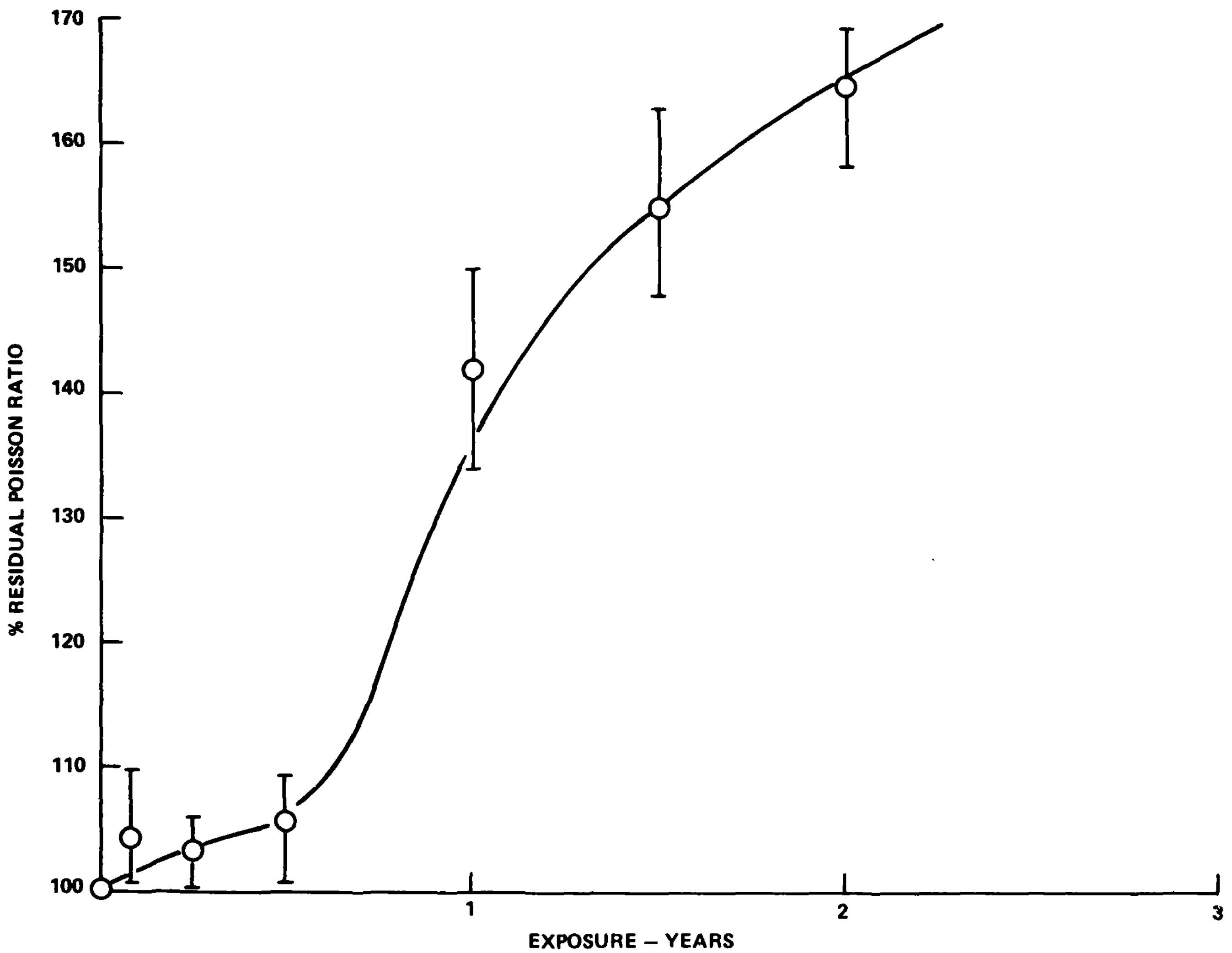


FIGURE 10 PERCENT CHANGE IN POISSON'S RATIO AS A RESULT OF EXPOSURE TIME IN PANAMA (200 SERIES UNCOATED)

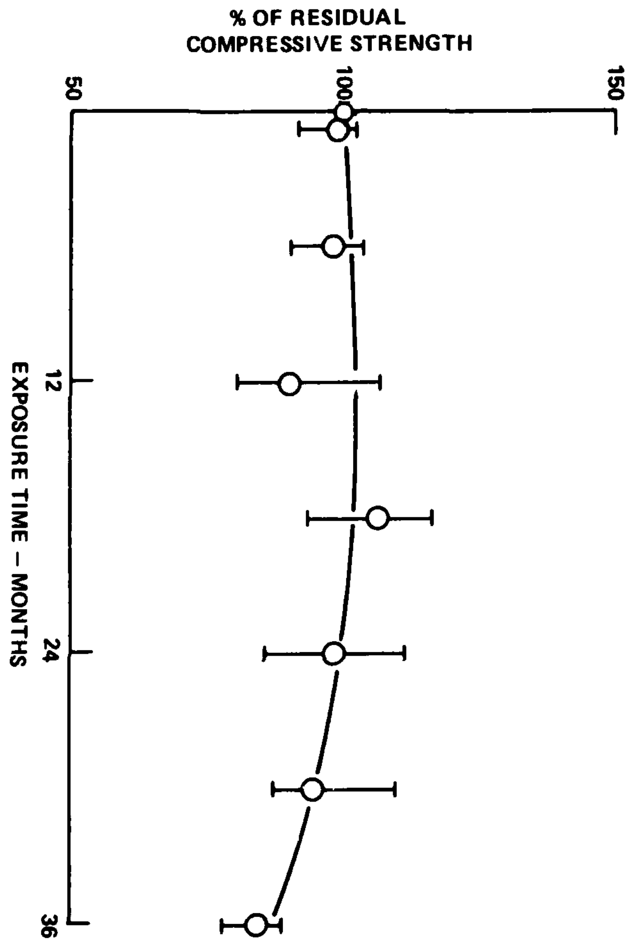


FIGURE 11 RESIDUAL COMPRESSIVE STRENGTH AS A FUNCTION OF EXPOSURE TIME IN WARMINSTER (300 SERIES)

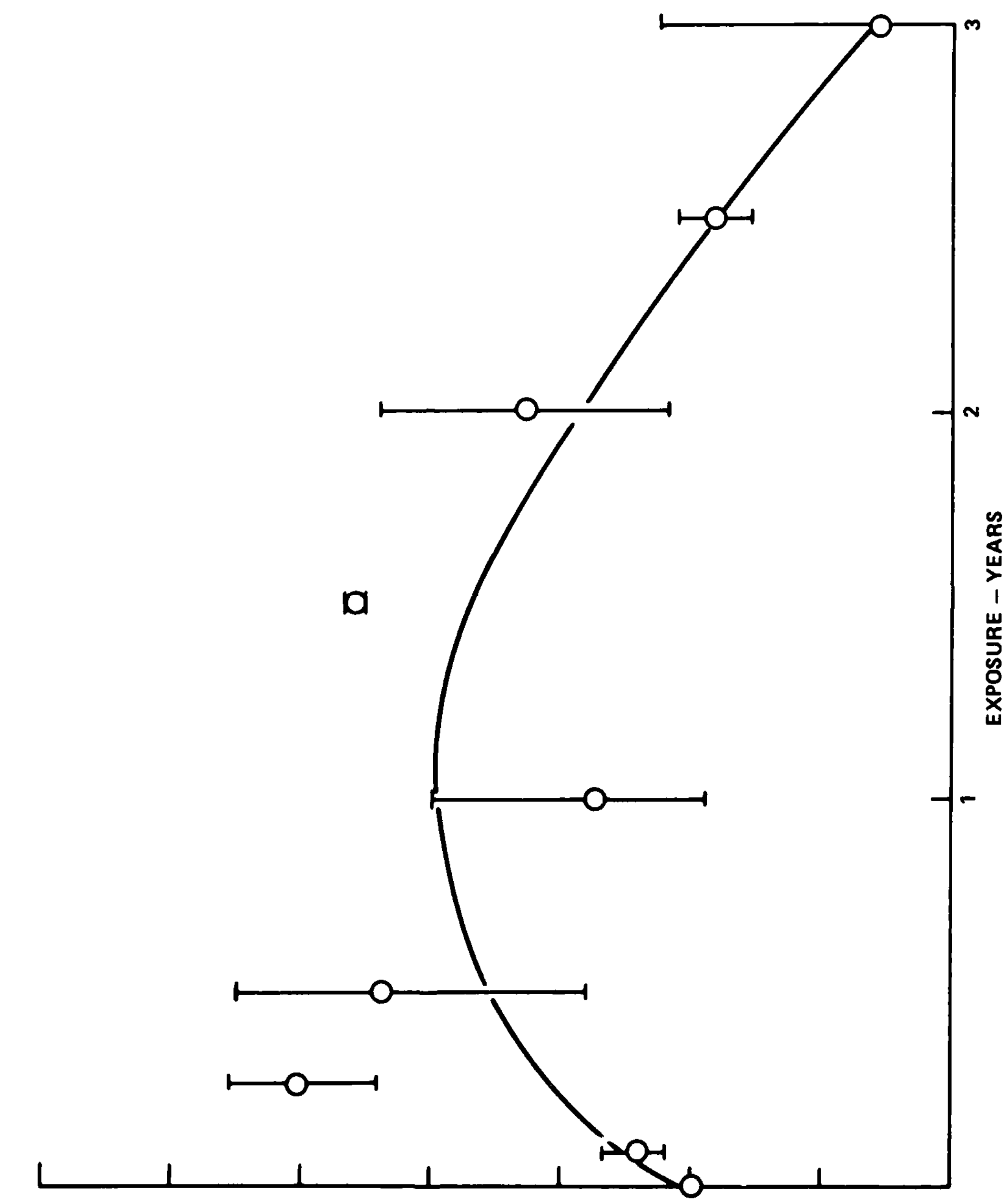


FIGURE 12 RESIDUAL COMPRESSIVE STRENGTH AS A FUNCTION OF EXPOSURE TIME IN WARMINSTER (UNCOATED 400 SERIES)

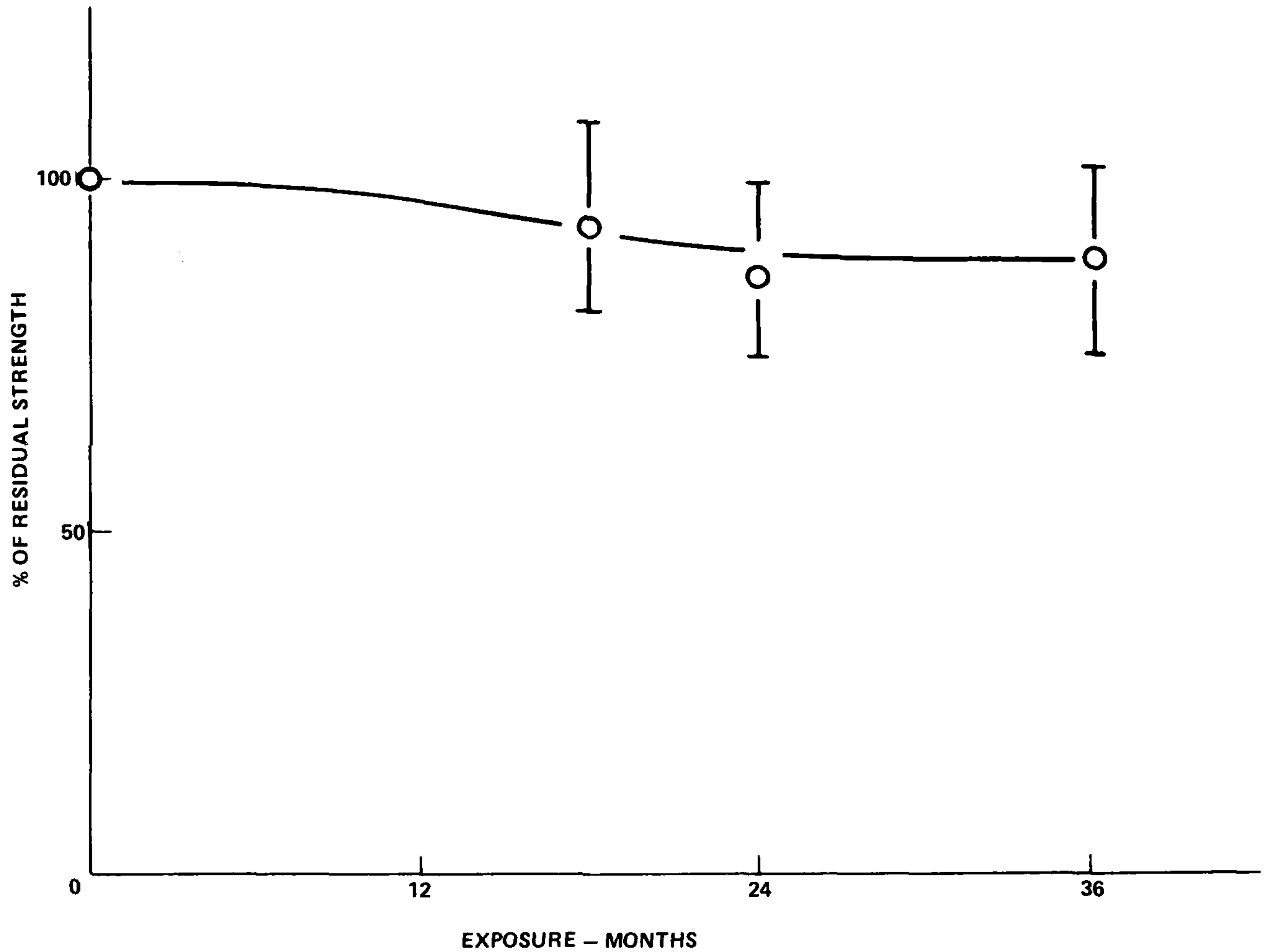


FIGURE 13. Room Temperature Compressive Strength as a Function of Exposure Time in Warminster (uncoated 700 series)

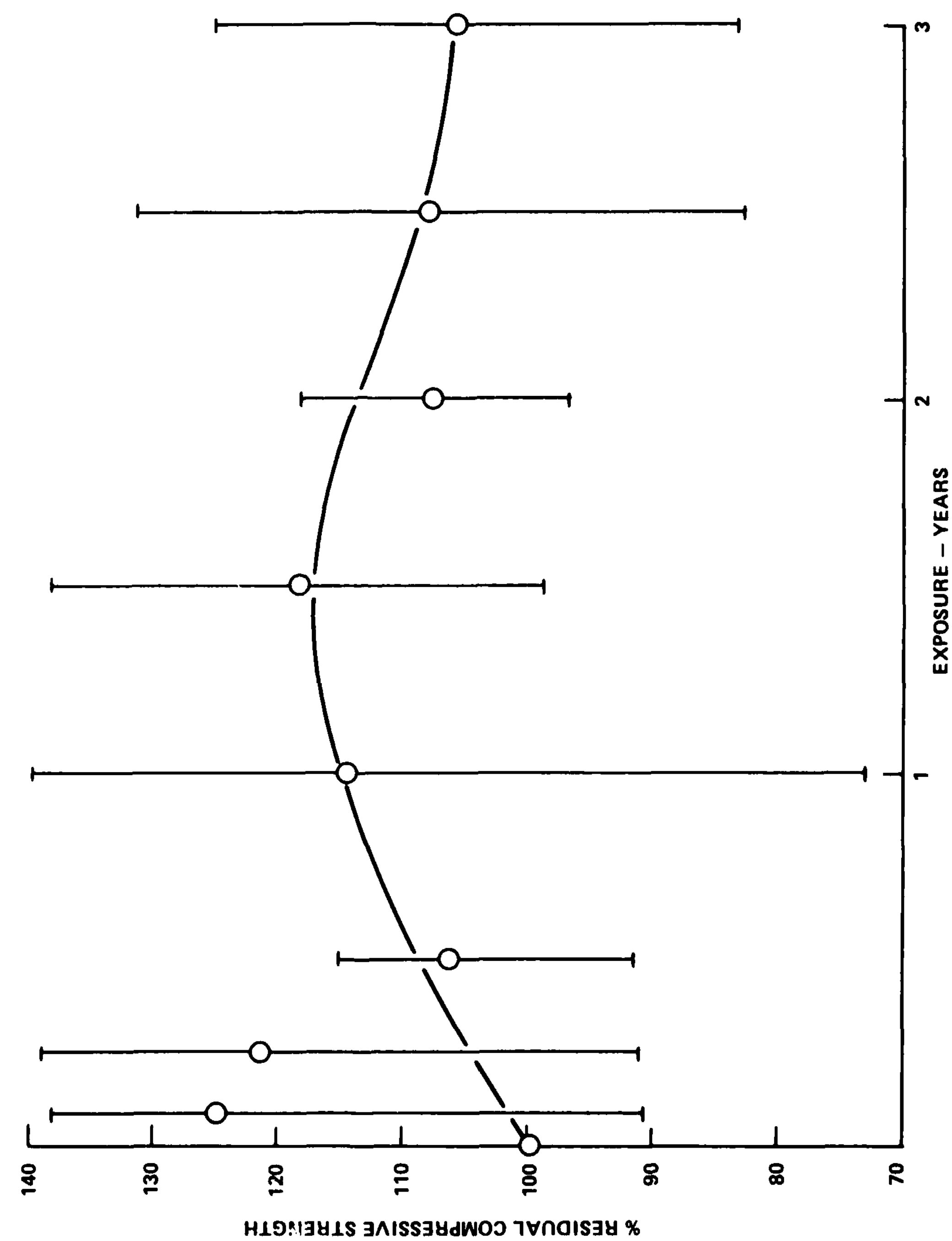


FIGURE 14 RESIDUAL COMPRESSIVE STRENGTH VERSUS EXPOSURE TIME IN WARMINSTER (COATED 400 SERIES)

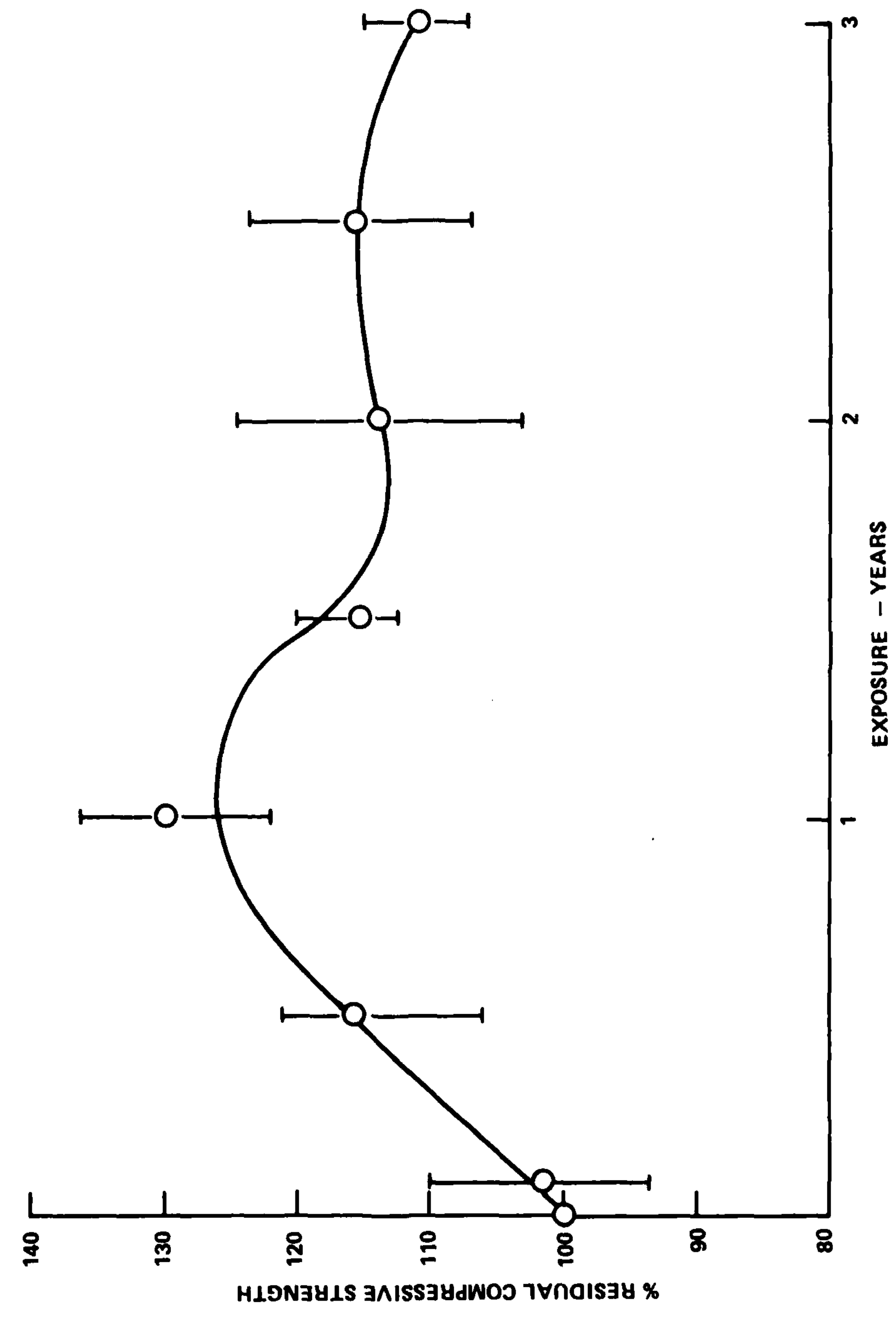


FIGURE 15. Residual Compressive Strength as a Function of Exposure Time in Warminster (coated 500 series)

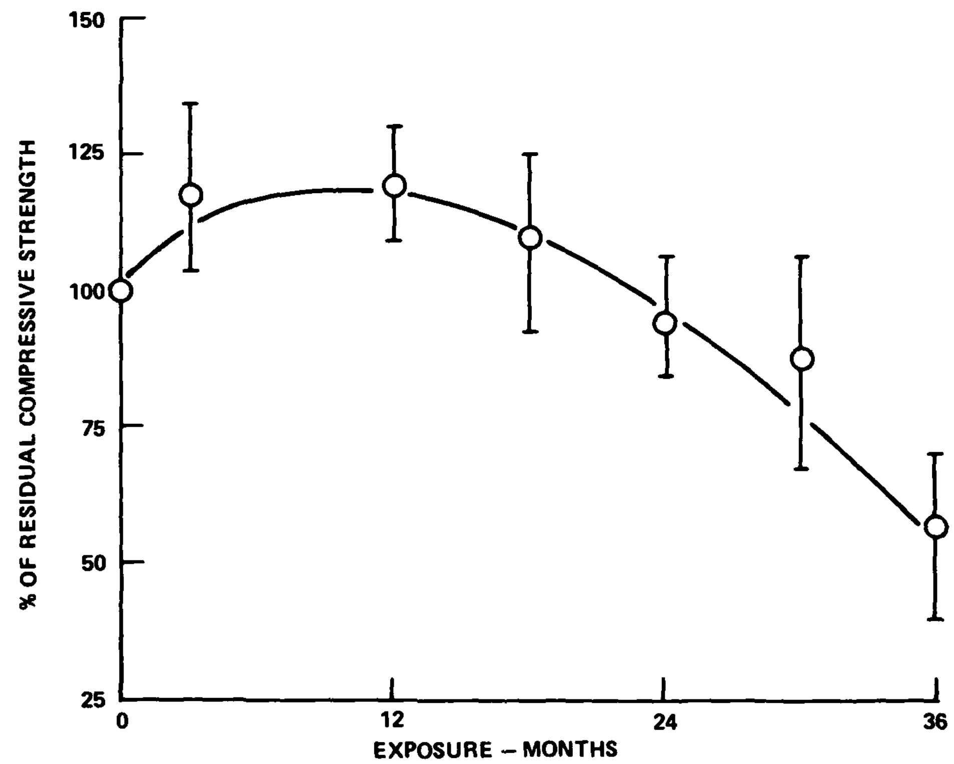


FIGURE 16. Residual Compressive Strength as a Function of Exposure Time in Panama (uncoated 200 series)

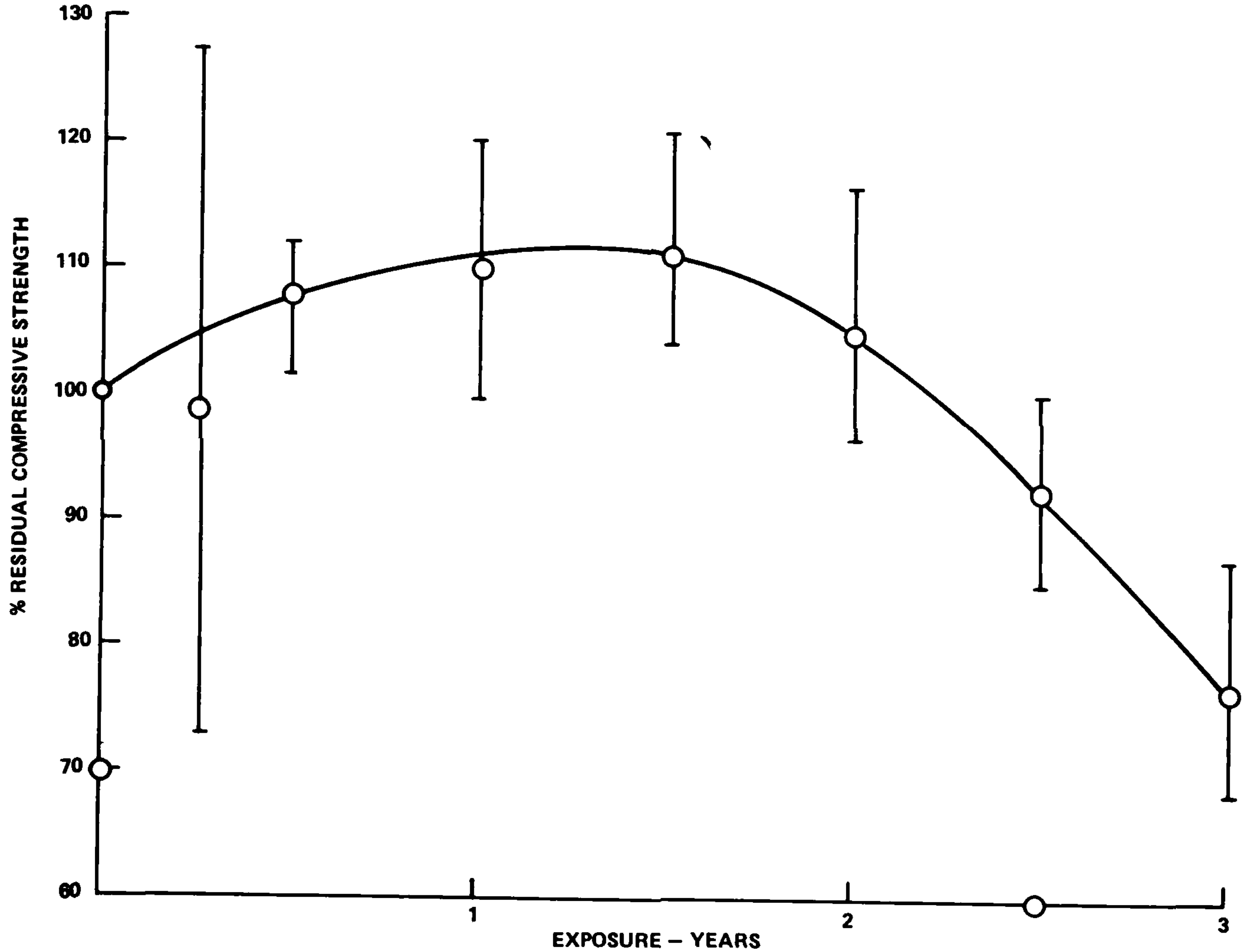


FIGURE 17 RESIDUAL COMPRESSIVE STRENGTH AS A FUNCTION OF EXPOSURE TIME IN PANAMA (UNCOATED 500 SERIES)

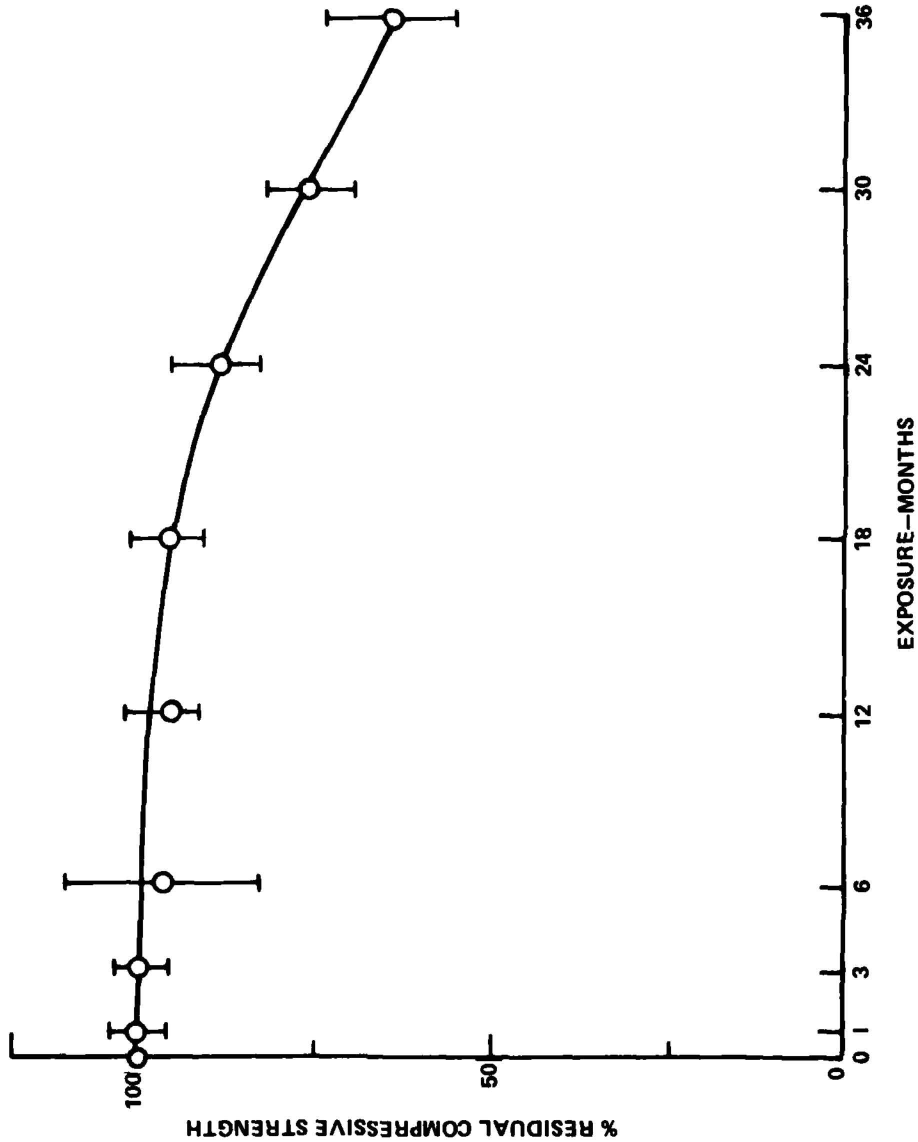


FIGURE 18. Room Temperature Compressive Strength as a Function of Exposure Time in Panama (uncoated 400 series)

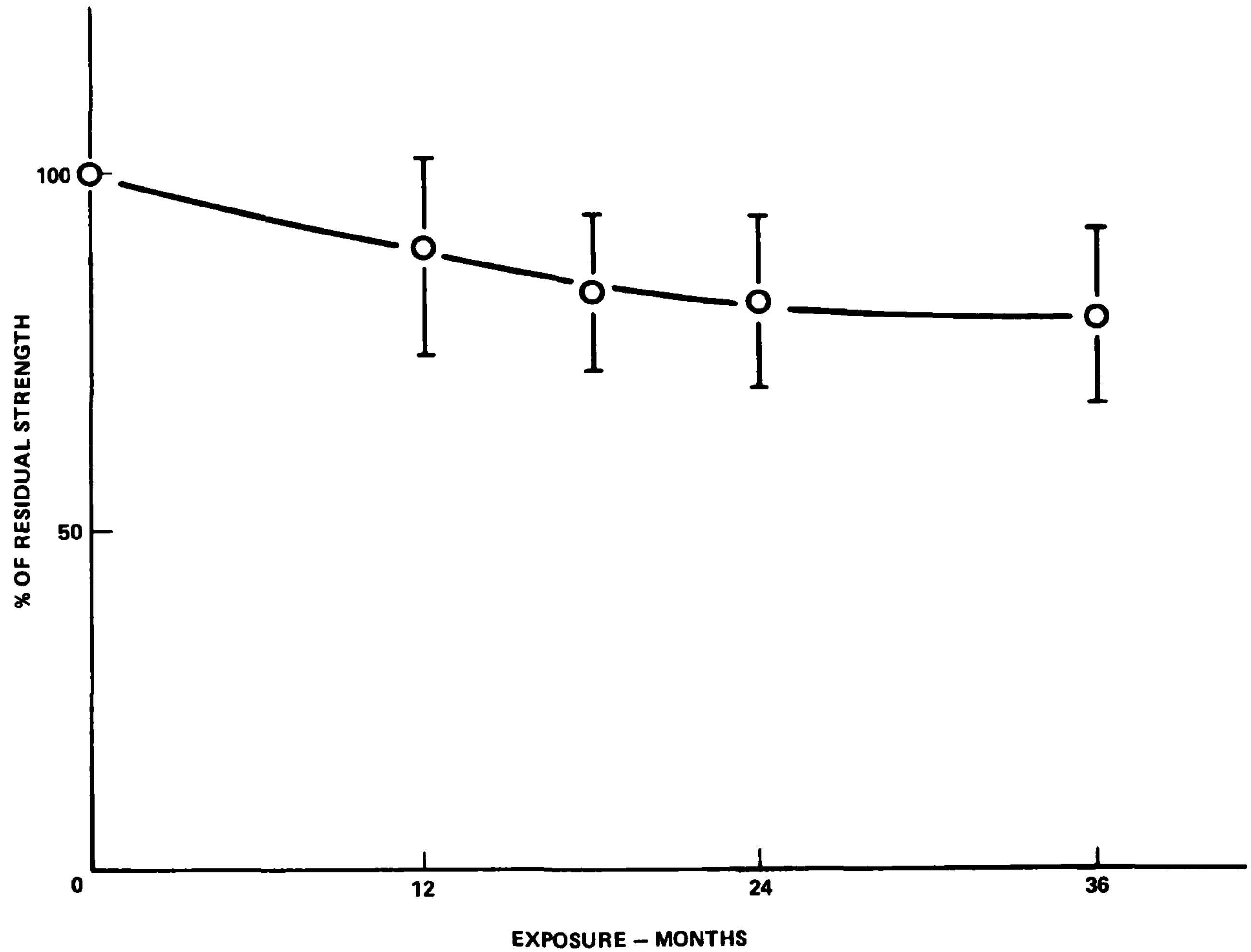


FIGURE 19. 250° F Residual Compressive Strength as a Function of Exposure Time in Warminster (uncoated 700 series)

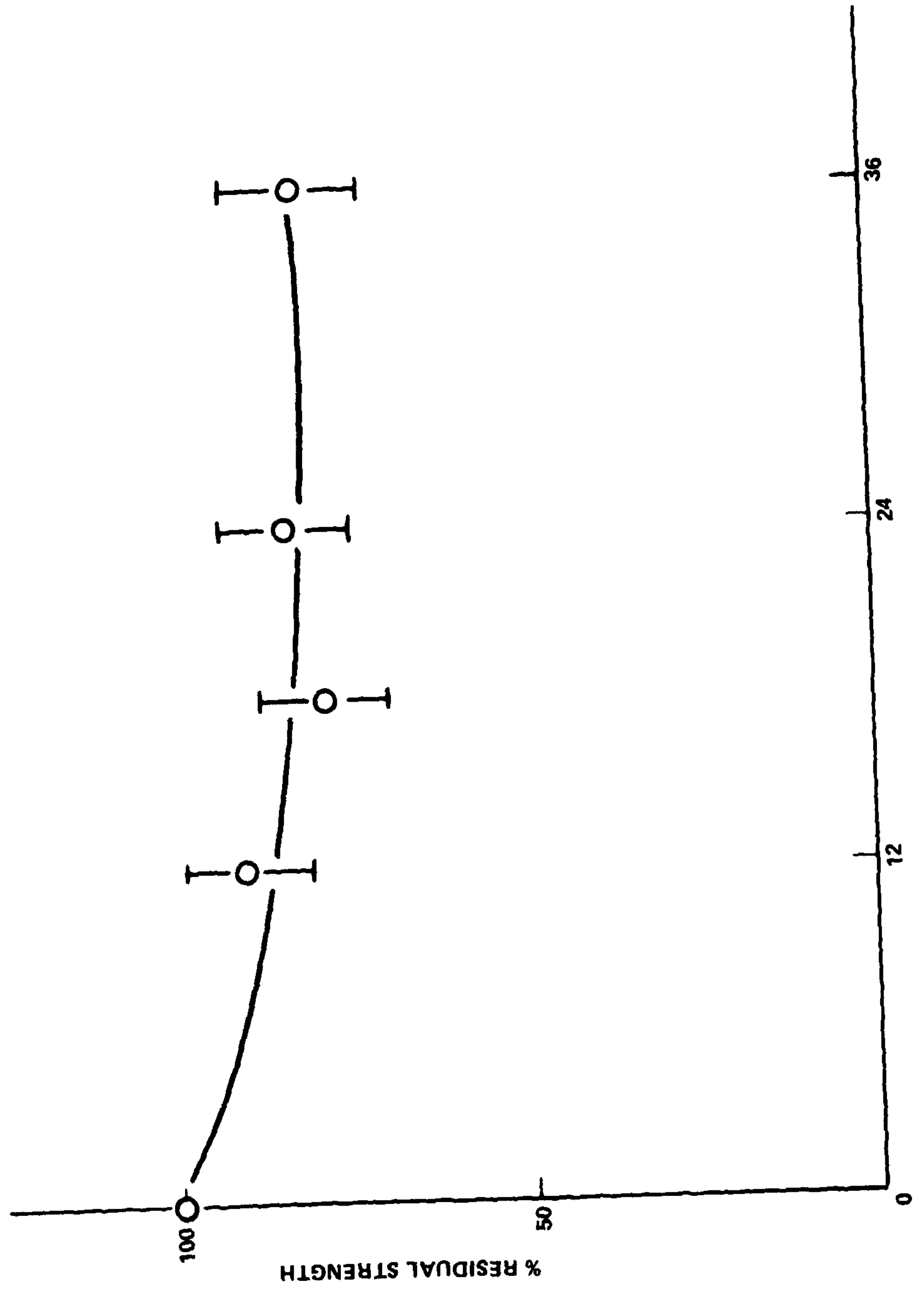


FIGURE 20. 250° F Residual Compressive Strength as a Function of Exposure Time in Panama (coated 700 series)

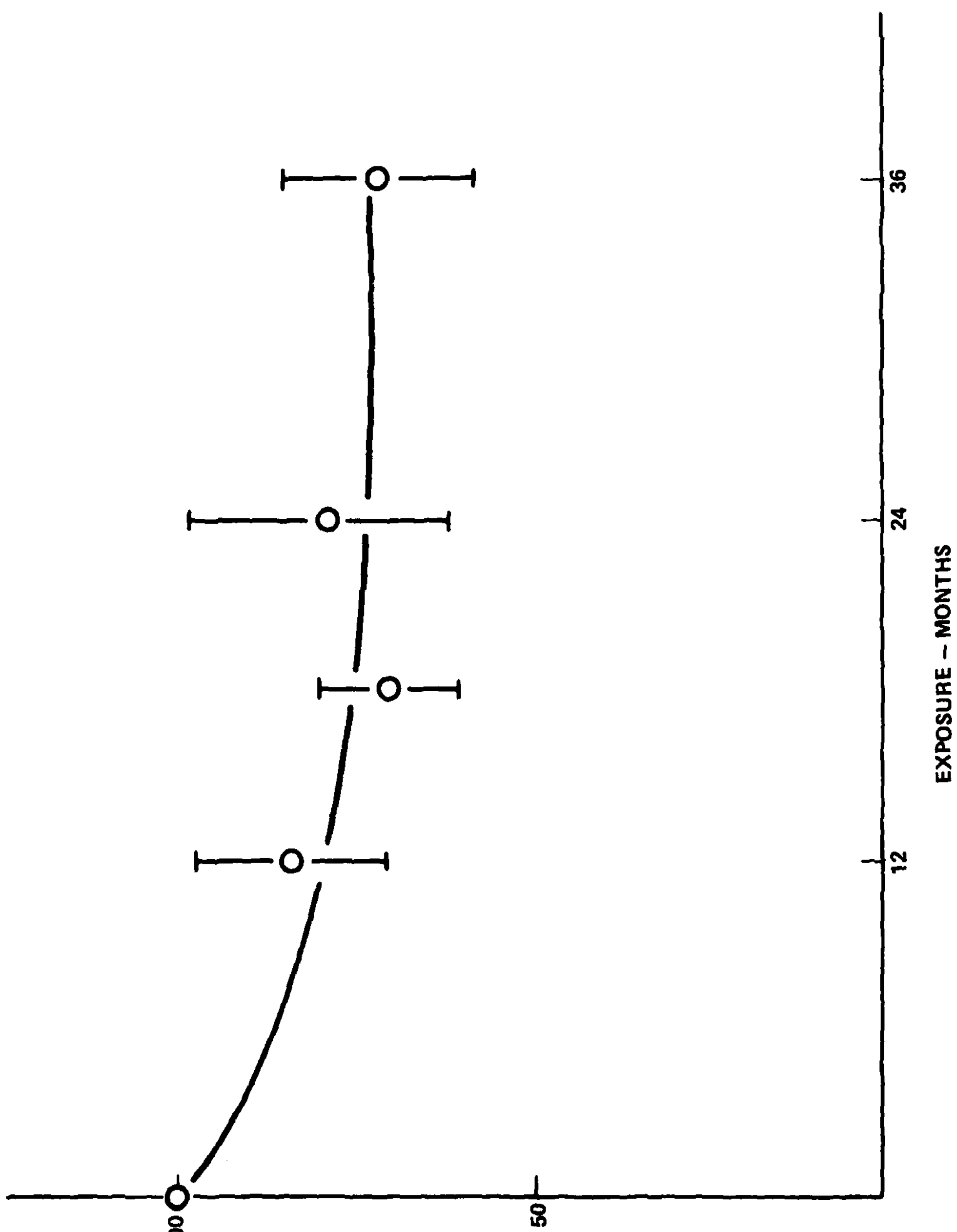


FIGURE 21. 250° F Residual Compressive Strength as a Function of Exposure Time in Panama (uncoated 700 series)

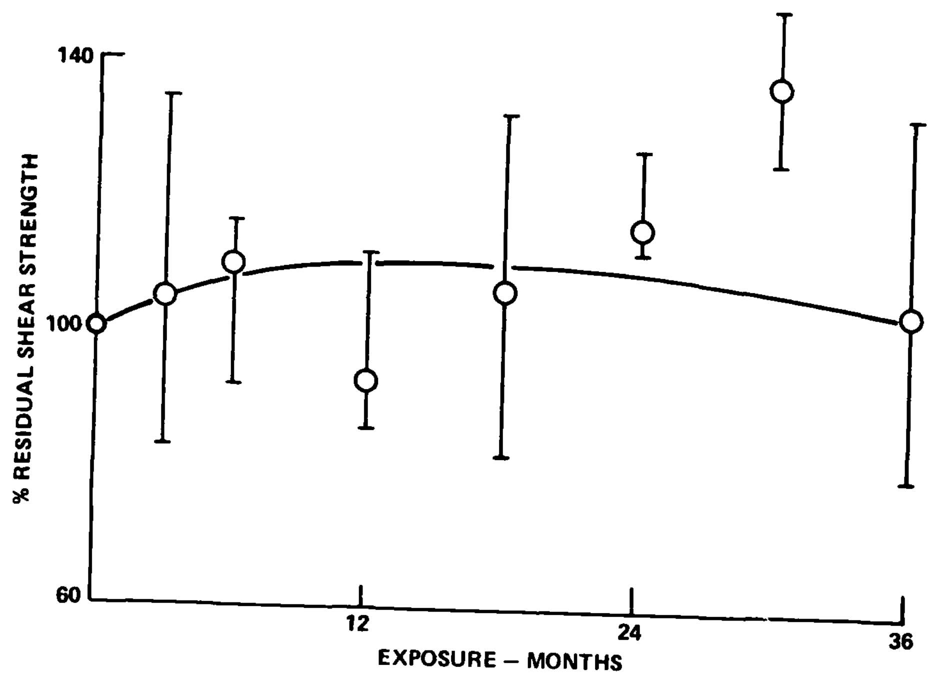


FIGURE 22. Residual Shear Strength as a Function of Exposure Time in Warminster (uncoated 400 series)

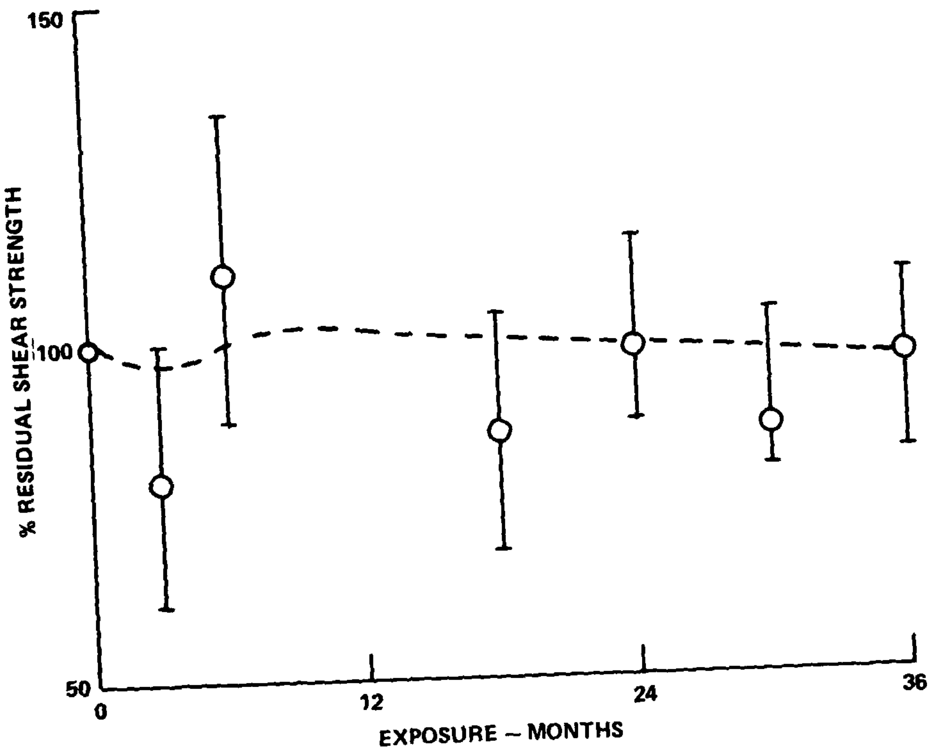


FIGURE 23. Residual Shear Strength as a Function of Exposure Time in Warminster (uncoated 500 series)

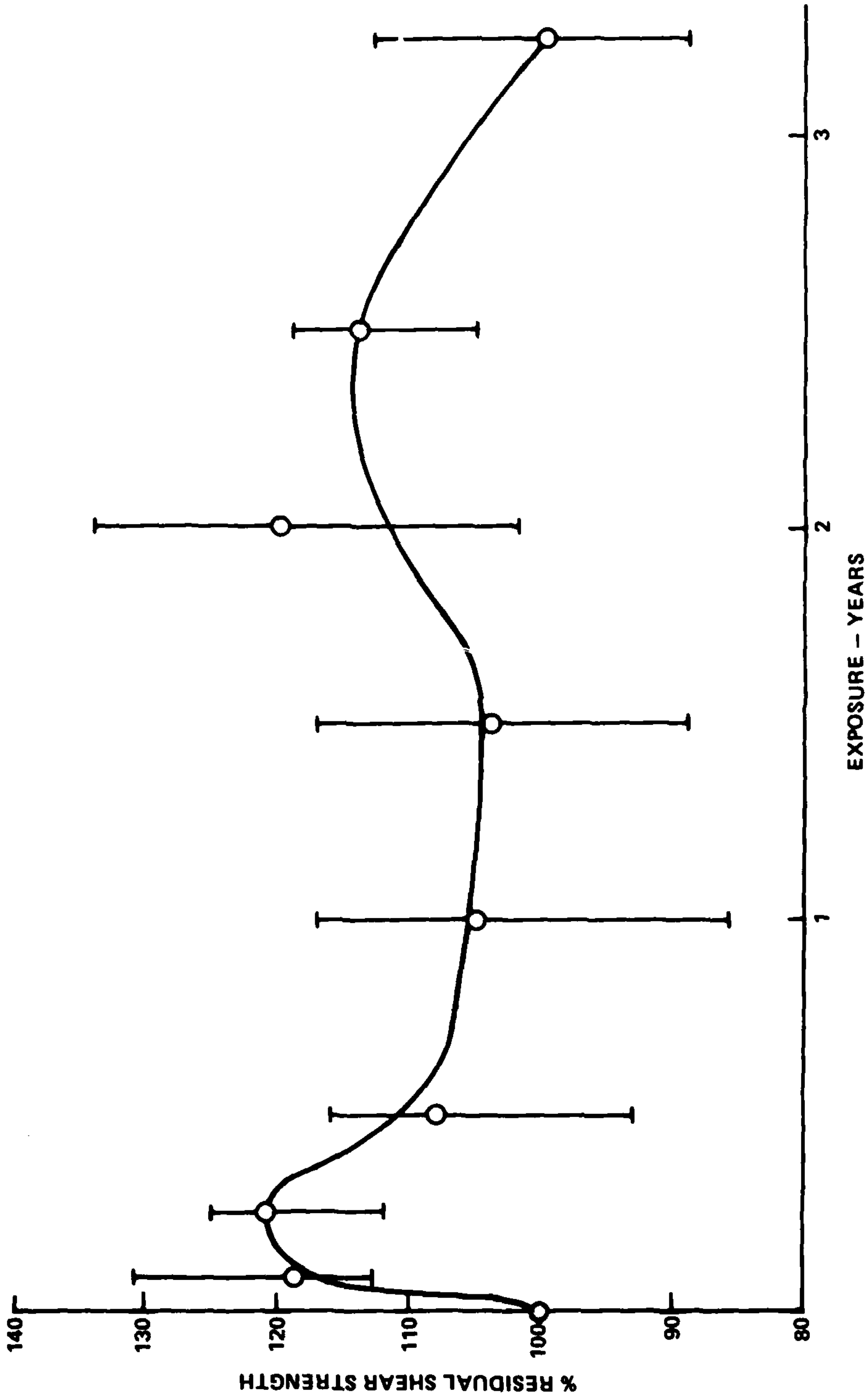


FIGURE 24. Residual Shear Strength as a Function of Exposure Time in Warminster (coated 100 series)

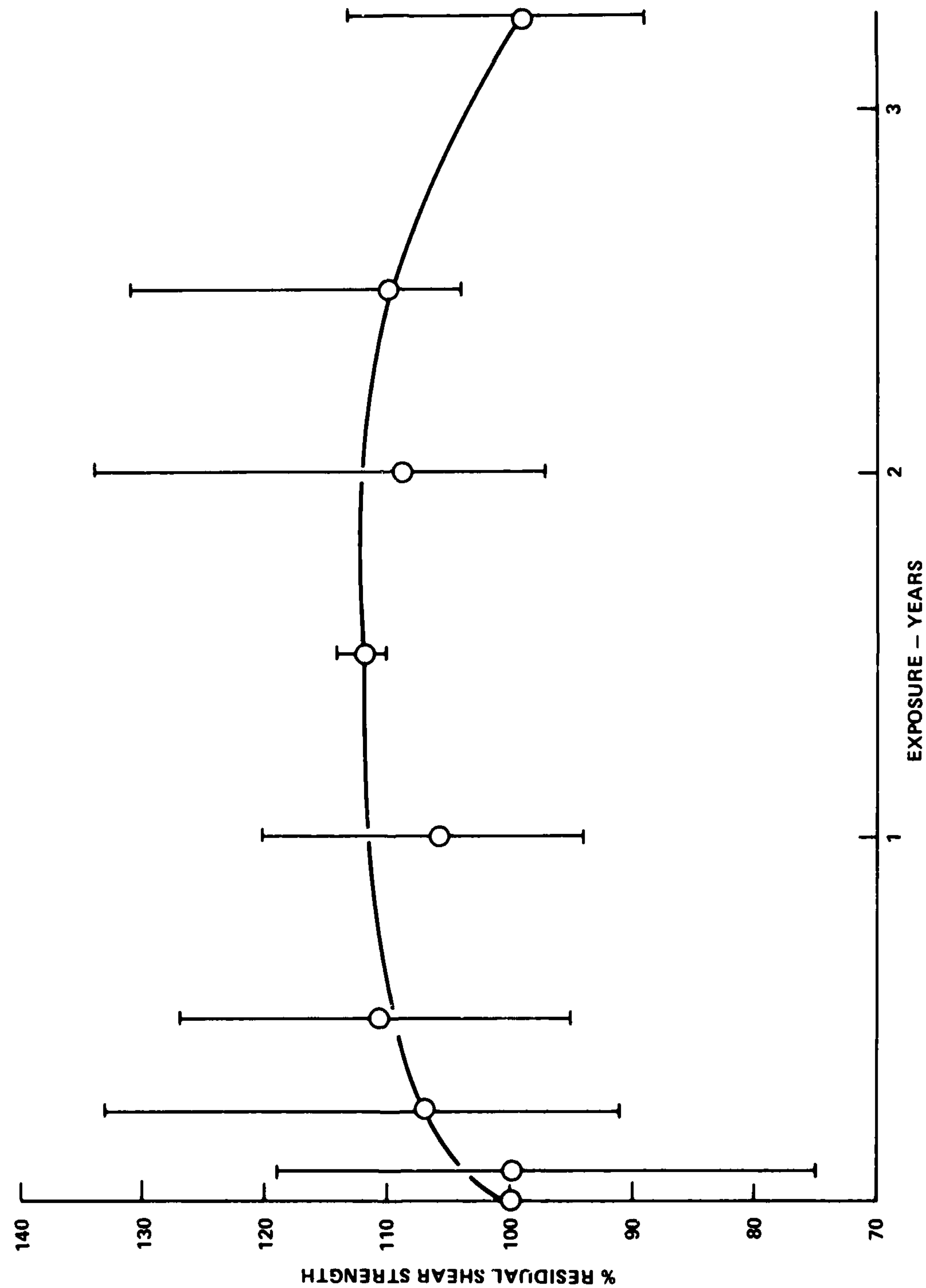


FIGURE 25. Residual Shear Strength as a Function of Exposure Time in Warminster (uncoated 100 series)

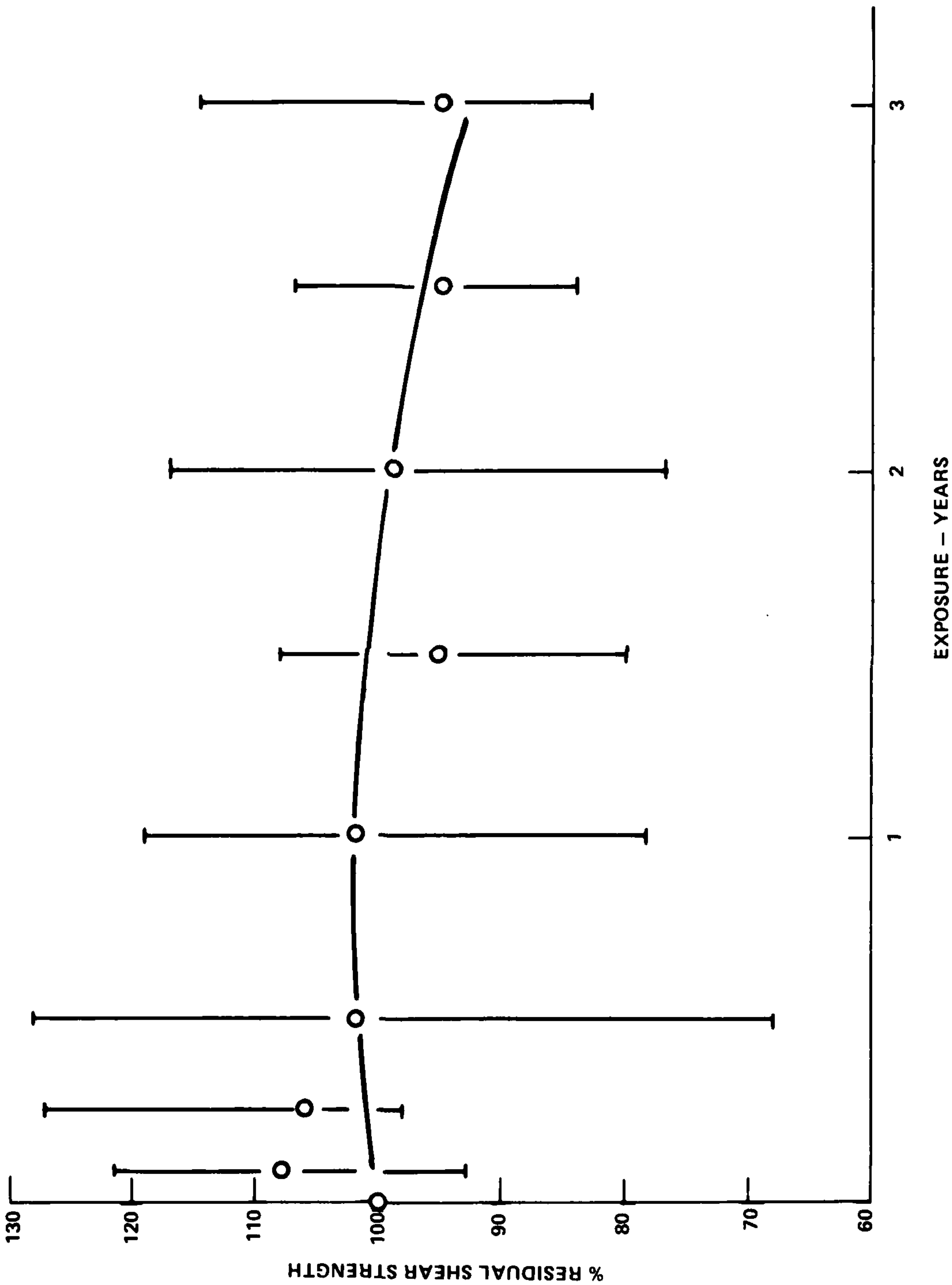


FIGURE 26. Residual Shear Strength as a Function of Exposure Time in Panama (uncoated 300 series)

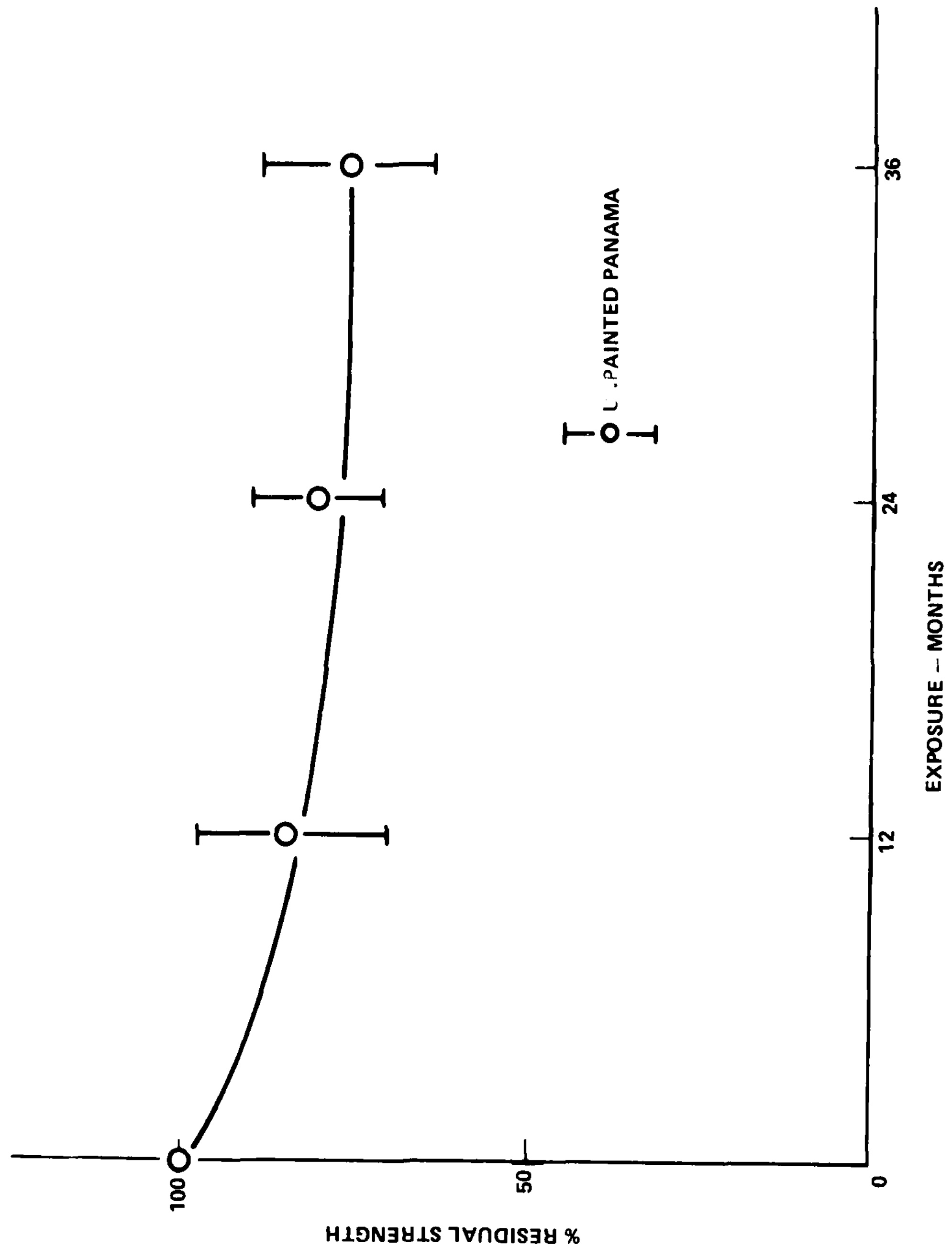


FIGURE 27. 250° F Residual Shear Strength as a Function of Exposure Time in Panama (uncoated 700 series)

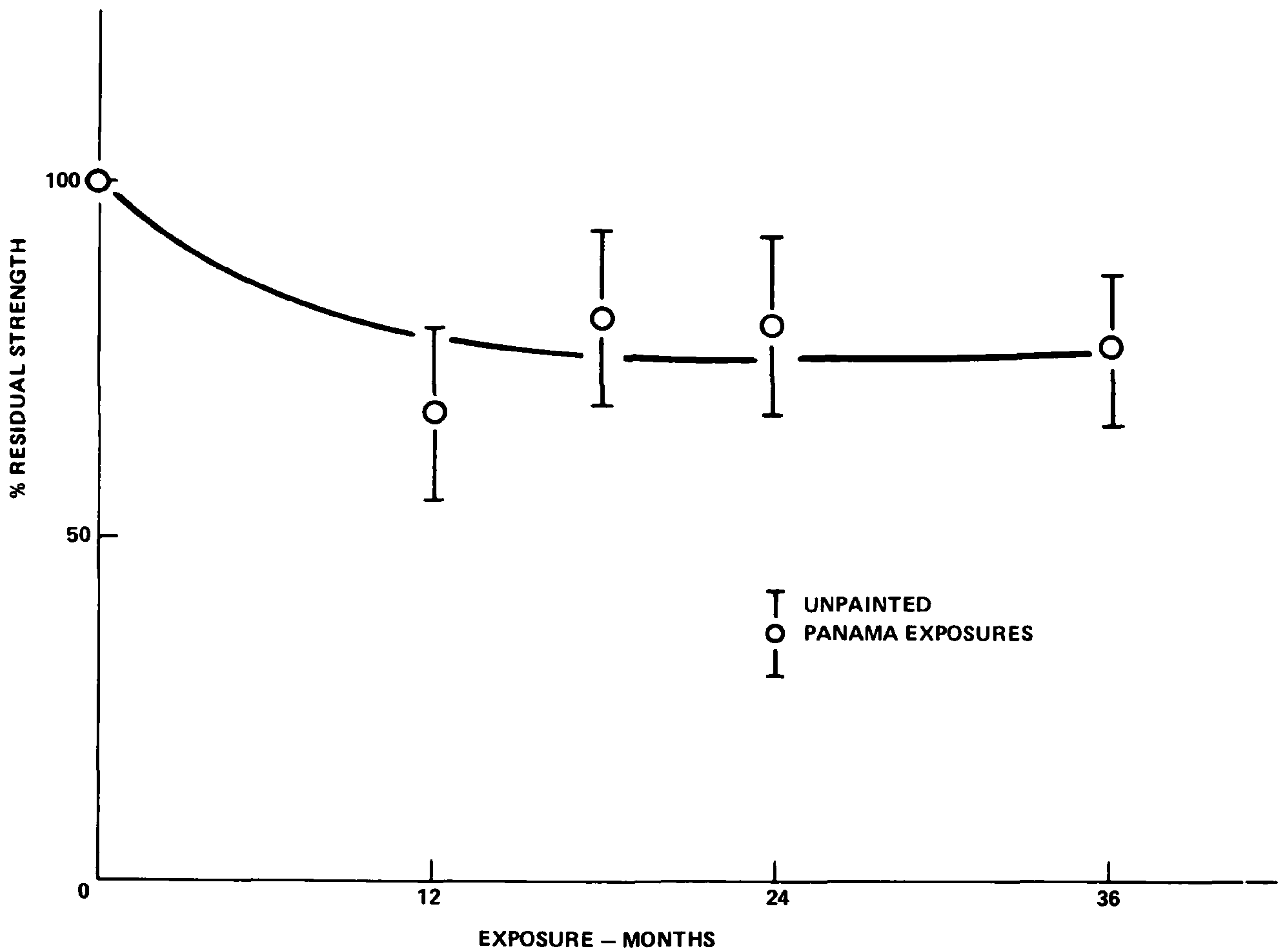


FIGURE 28. 250° F Residual Flexure Strength as a Function of Exposure Time in Panama (uncoated 700 series)

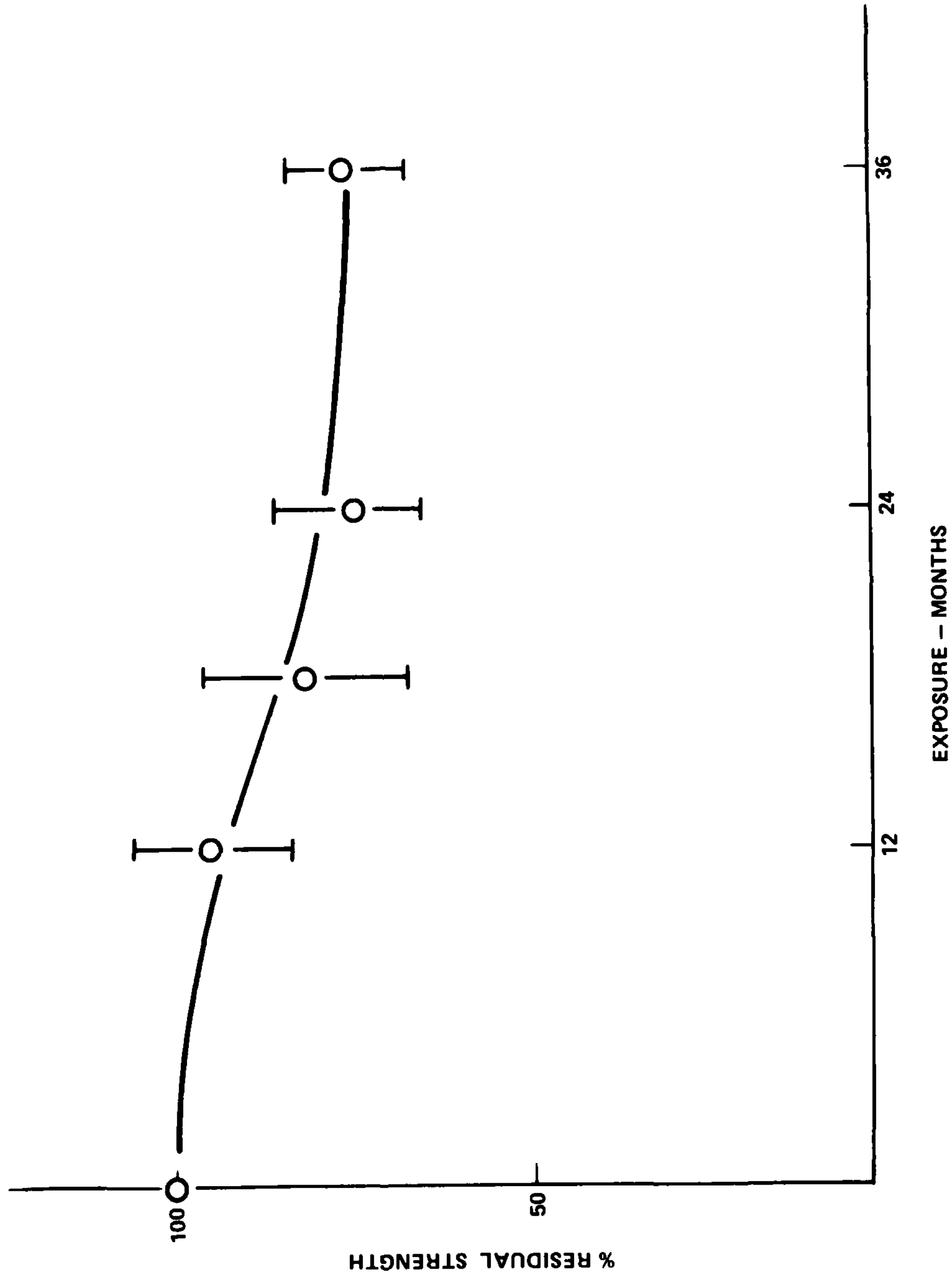


FIGURE 29. 250° F Residual Flexure Strength as a Function of Exposure Time in Panama (coated 700 series)

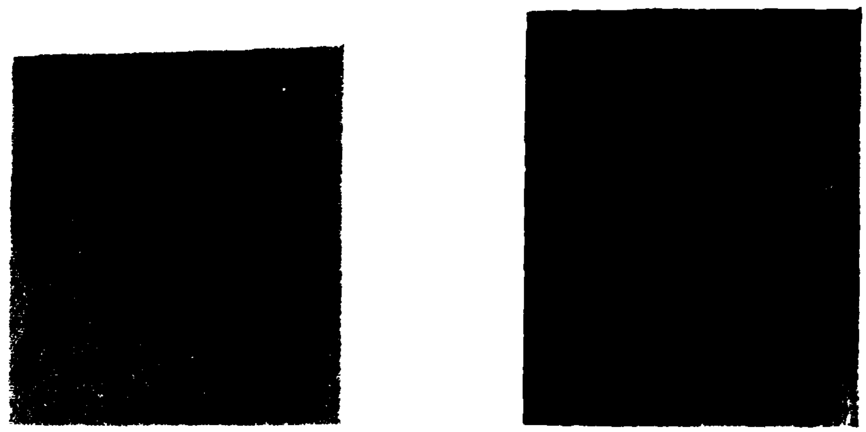
UNEXPOSED 36 MONTHS EXPOSURE

(a) 200 SERIES

(b) 500 SERIES

FIGURE 30 A COMPARISON OF SURFACE CONDITIONS OF UNPAINTED SPECIMENS BEFORE AND AFTER 36 MONTHS EXPOSURE IN PANAMA (2X)

UNEXPOSED   36 MONTHS EXPOSURE

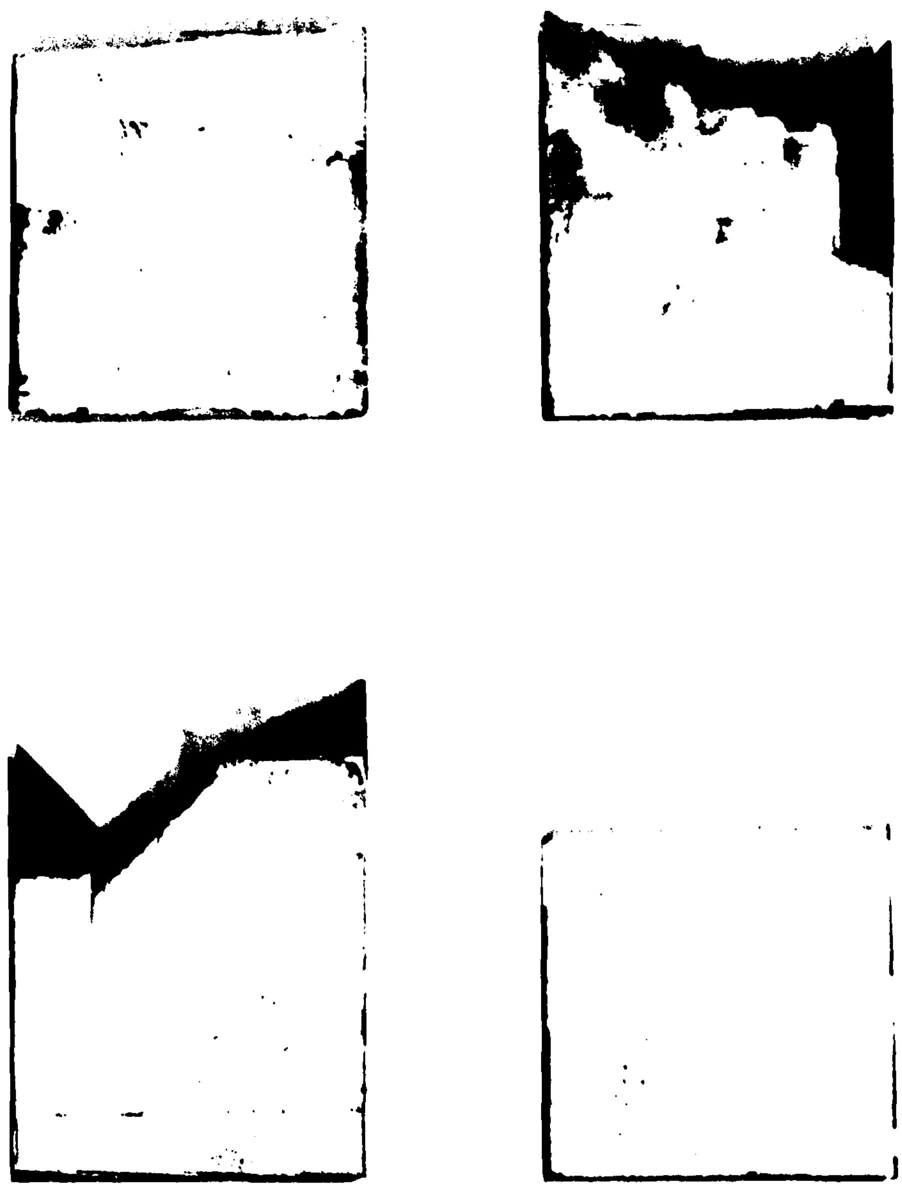


(a) 400 SERIES

(b) 700 SERIES

FIGURE 31 A COMPARISON OF SURFACE CONDITIONS OF UNPAINTED SPECIMENS BEFORE AND AFTER 36 MONTHS EXPOSURE IN PANAMA (2X)

UNEXPOSED 36 MONTHS EXPOSURE

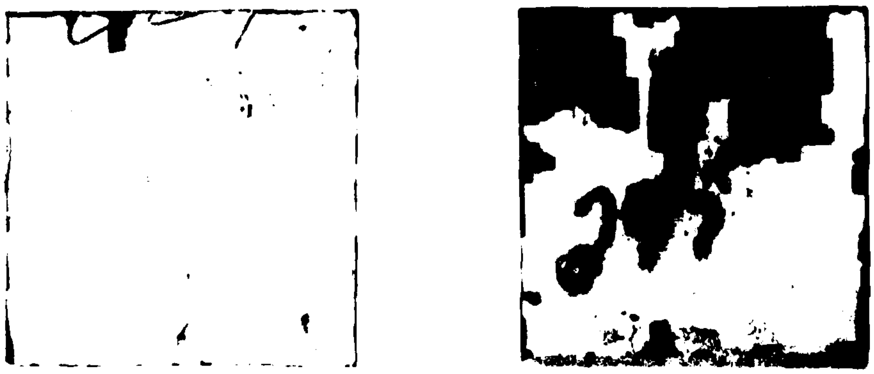


(a) 100 SERIES

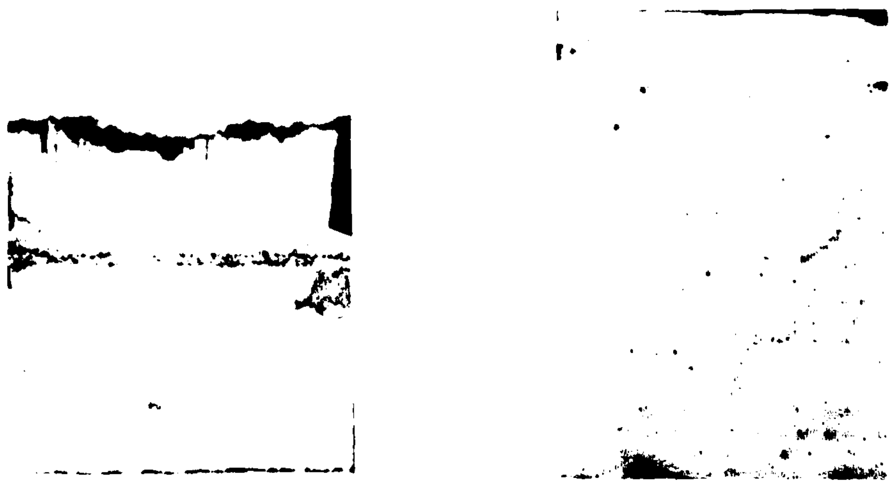
(b) 700 SERIES

FIGURE 32 A COMPARISON OF SURFACE CONDITIONS OF COATED SPECIMENS BEFORE & AFTER 36 MONTHS EXPOSURE IN PANAMA (2X)

UNEXPOSED 36 MONTHS EXPOSURE



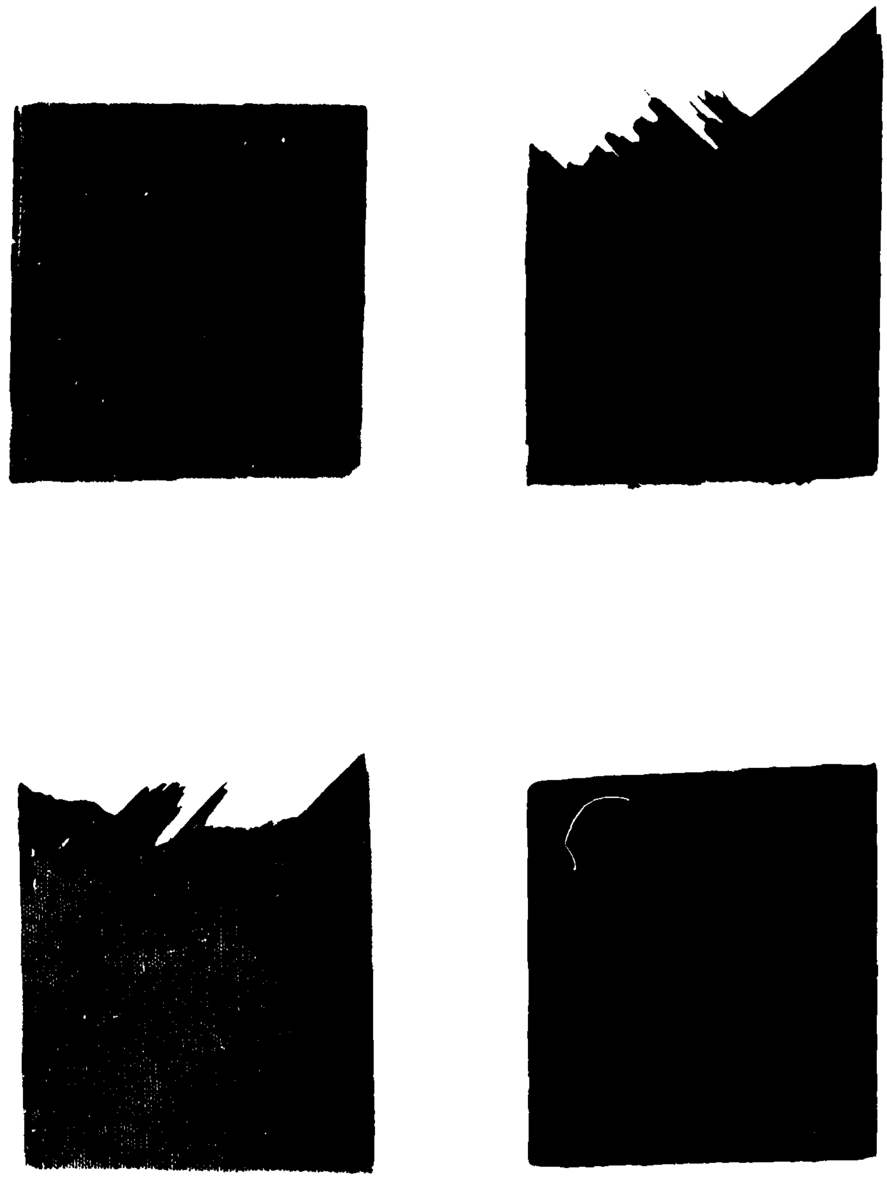
(a) 200 SERIES



(b) 700 SERIES

FIGURE 33 A COMPARISON OF SURFACE CONDITIONS OF COATED SPECIMENS BEFORE AND AFTER 36 MONTHS EXPOSURE IN PANAMA (2X)

UNEXPOSED — 36 MONTHS EXPOSURE



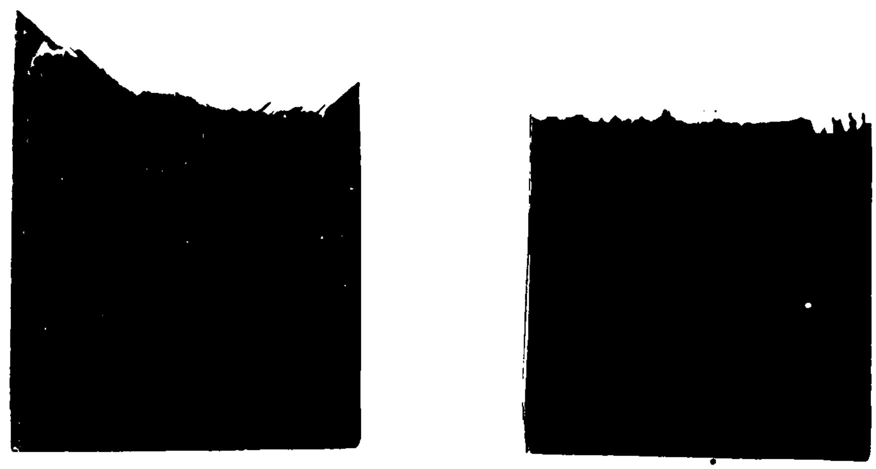
(a) 200 SERIES

(b) 400 SERIES

FIGURE 34 A COMPARISON OF SURFACE CONDITIONS OF UNCOATED SPECIMENS BEFORE & AFTER 36 MONTHS EXPOSURE IN WARMINSTER (2X)

UNEXPOSED UNCOATED

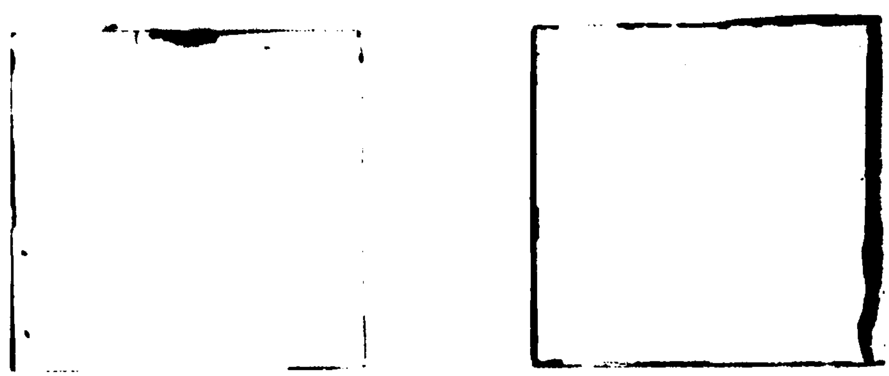
36 MONTHS EXPOSURE UNCOATED



(a) 700 SERIES

COATED

COATED



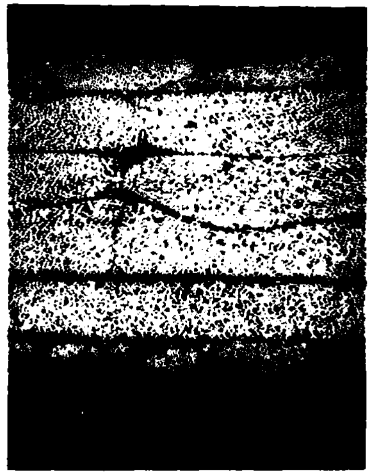
(b) 700 SERIES

FIGURE 35 A COMPARISON OF SURFACE CONDITIONS OF COATED & UNCOATED SPECIMENS OF 700 SERIES MATERIAL AFTER 36 MONTHS EXPOSURE IN WARMINSTER (2X)

UNEXPOSED

EXPOSED 36 MONTHS IN PANAMA

A. 100 SERIES MATERIAL



B. 200 SERIES MATERIAL

FIGURE 36 A COMPARISON OF UNCOATED EXPOSED AND UNEXPOSED LAMINATE MICROSTRUCTURES (35X)

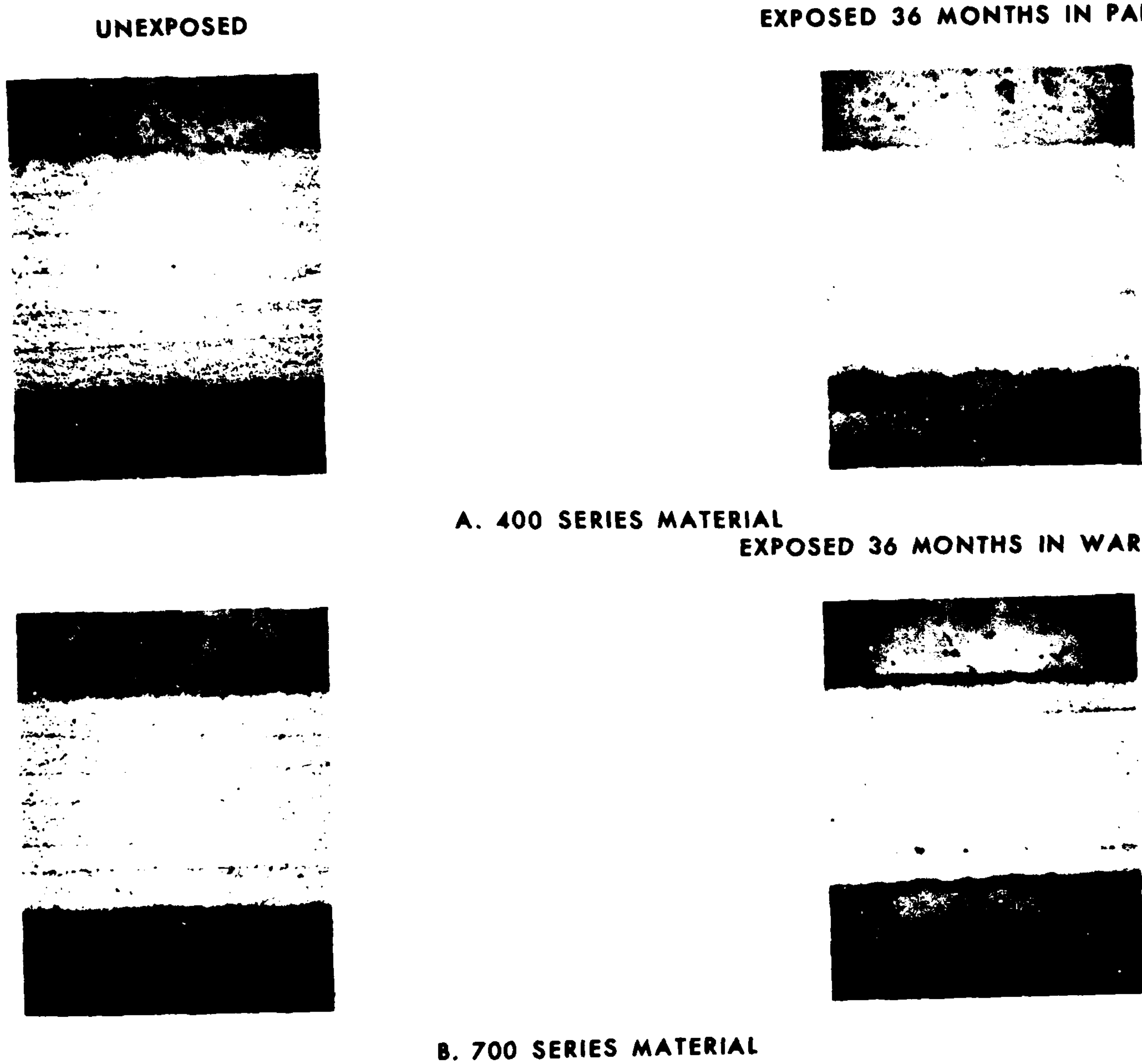
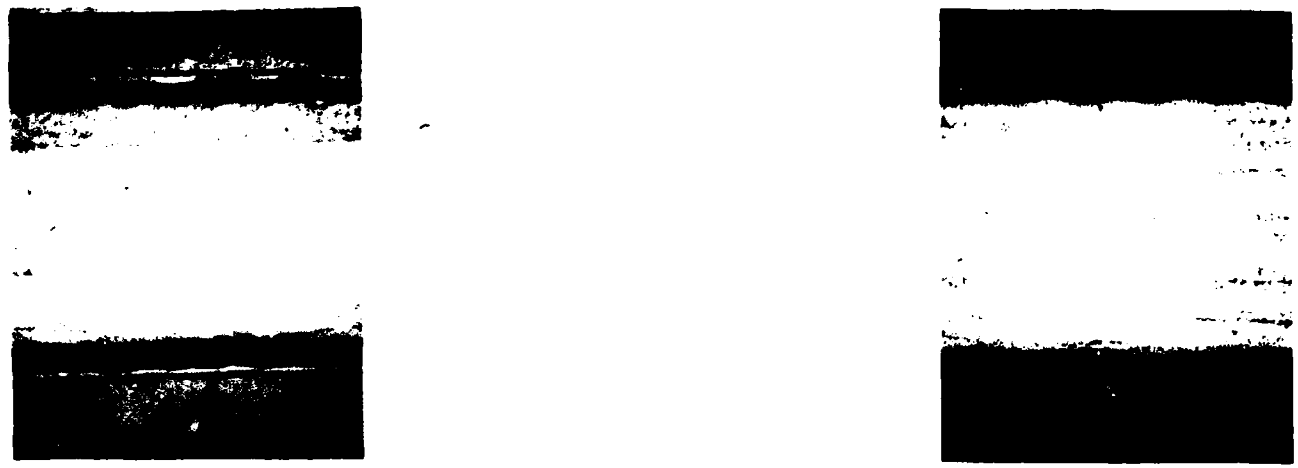


FIGURE 37 A COMPARISON OF UNCOATED EXPOSED AND UNEXPOSED LAMINATE MICROSTRUCTURES (35X)

UNEXPOSED EXPOSED 36 MONTHS IN PANAMA



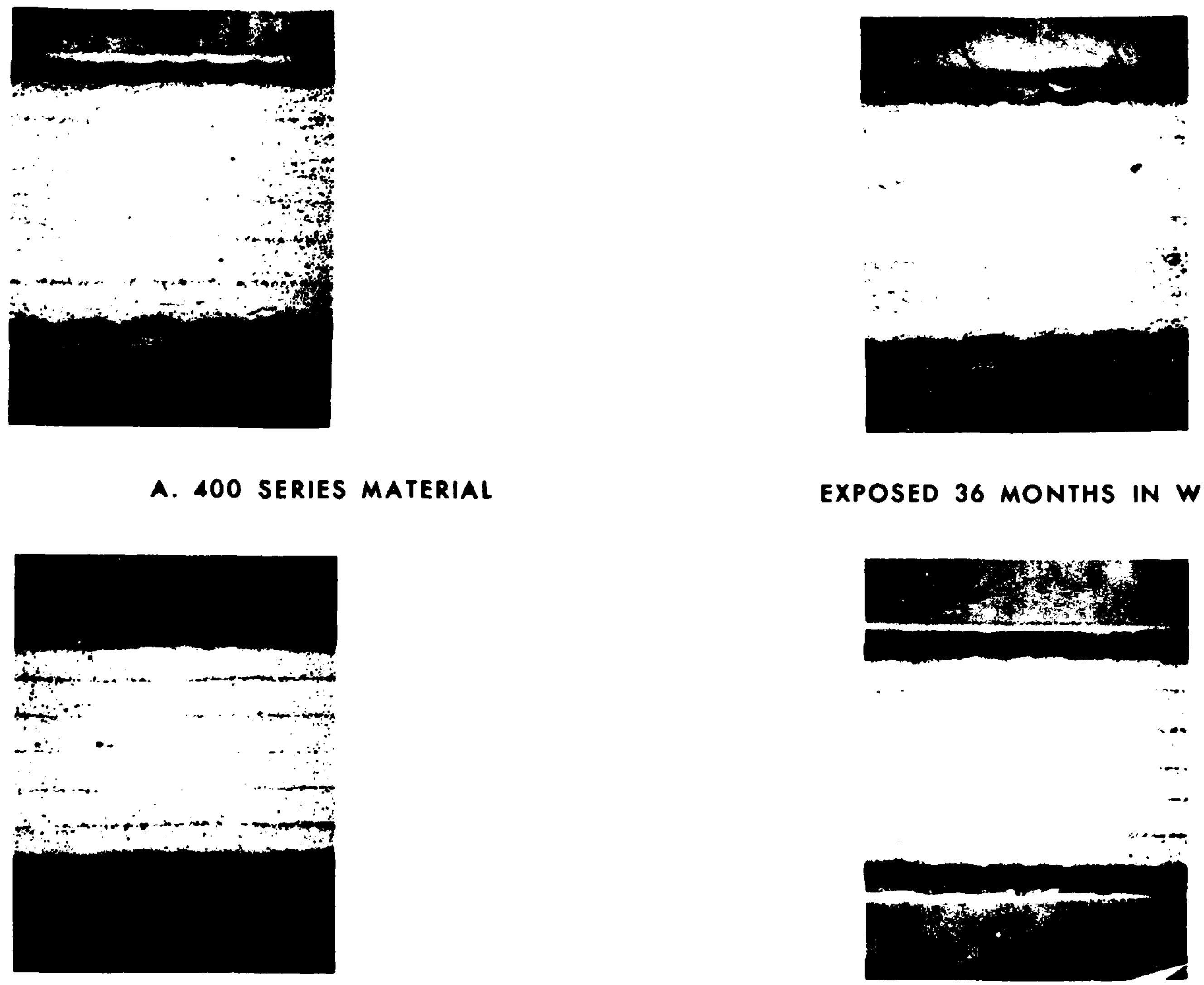
A. 100 SERIES MATERIAL

B. 200 SERIES MATERIAL

FIGURE 38 A COMPARISON OF COATED EXPOSED AND UNEXPOSED LAMINATE MICROSTRUCTURES (35X)

UNEXPOSED

EXPOSED 36 MONTHS IN PANAMA



A. 400 SERIES MATERIAL

EXPOSED 36 MONTHS IN WARMINSTER

B. 700 SERIES MATERIAL

FIGURE 39 A COMPARISON OF COATED EXPOSED AND UNEXPOSED LAMINATE MICROSTRUCTURES (35X)

FIGURE 40 A PHOTO MICROGRAPH DEPICTING THE LARGE RESIN RICH AREAS COMPLETELY BRIDGING THE 0° PLIES IN 200 SERIES MATERIAL EXPOSED FOR 24 MONTHS IN PANAMA

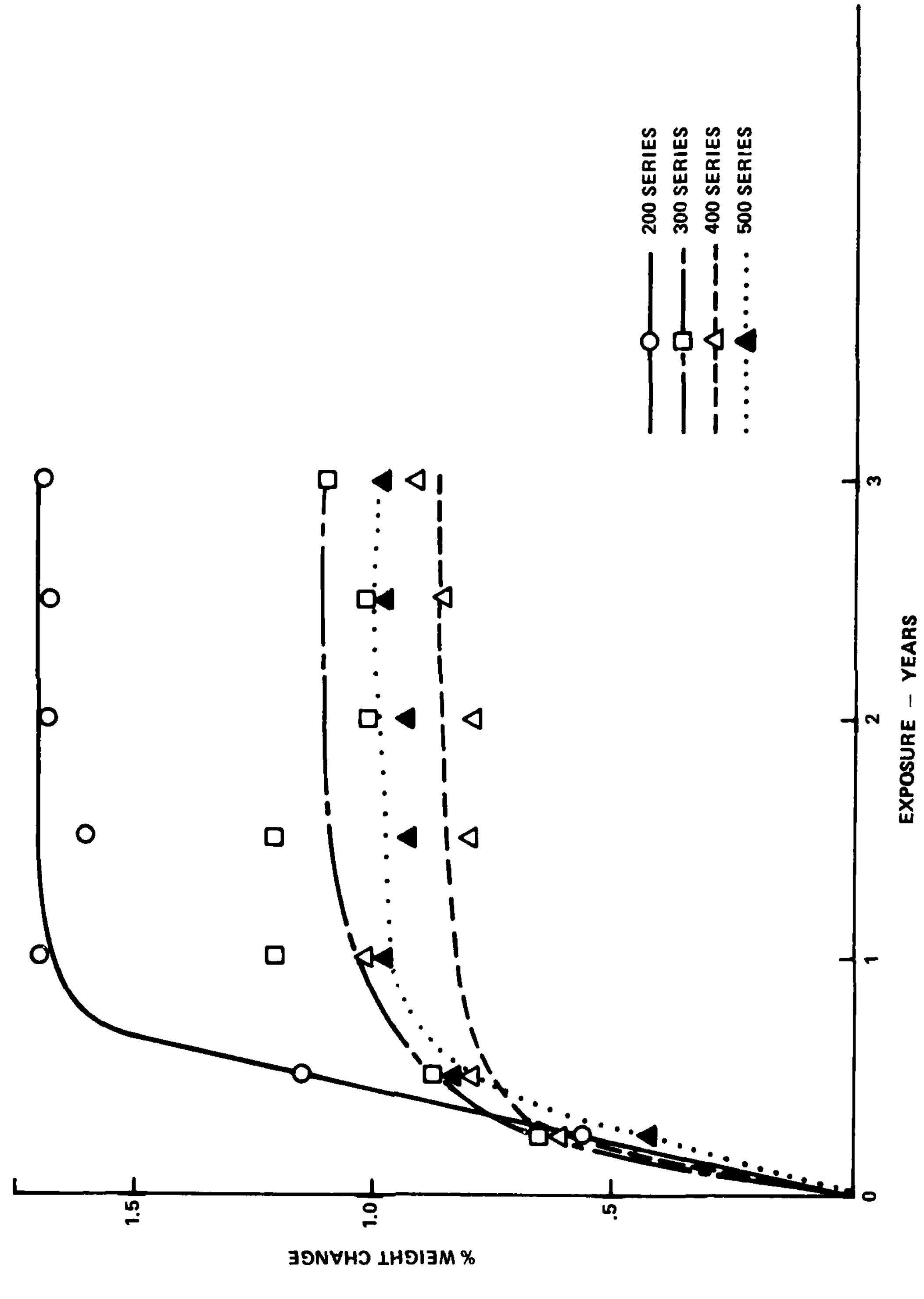


FIGURE 41 - Percent Weight Change for Painted Panels Exposed at Warminster

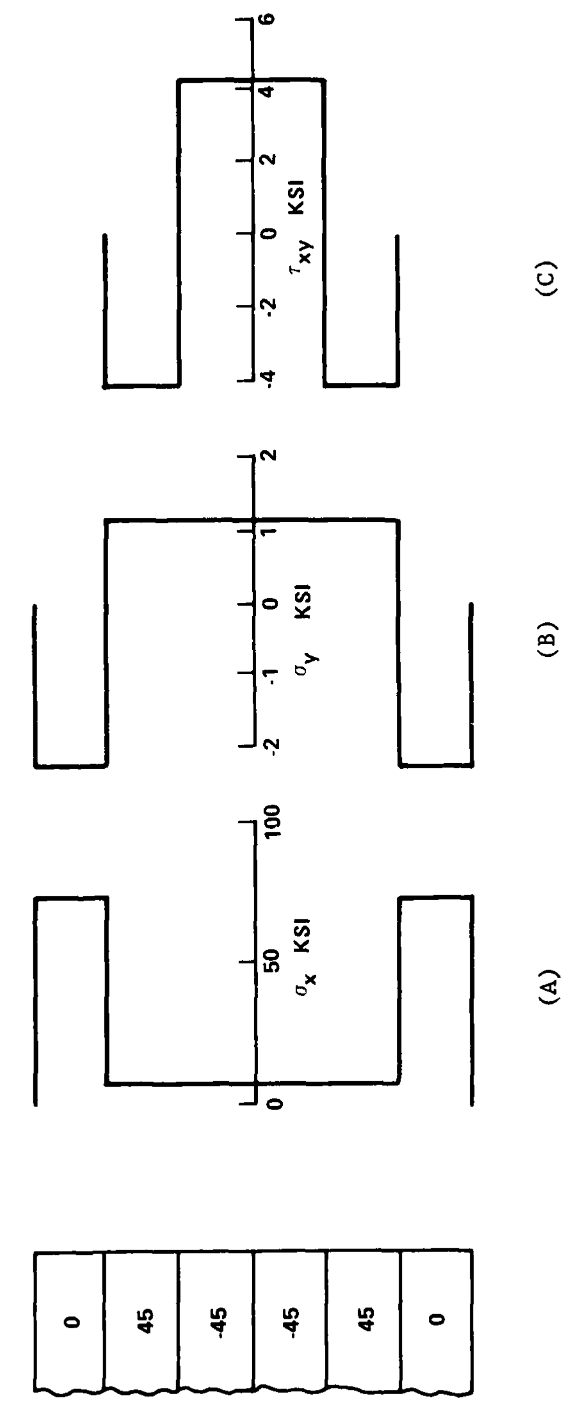


FIGURE 42. Stress Analysis for Tensile Loading Along 0° Plies (100 series)

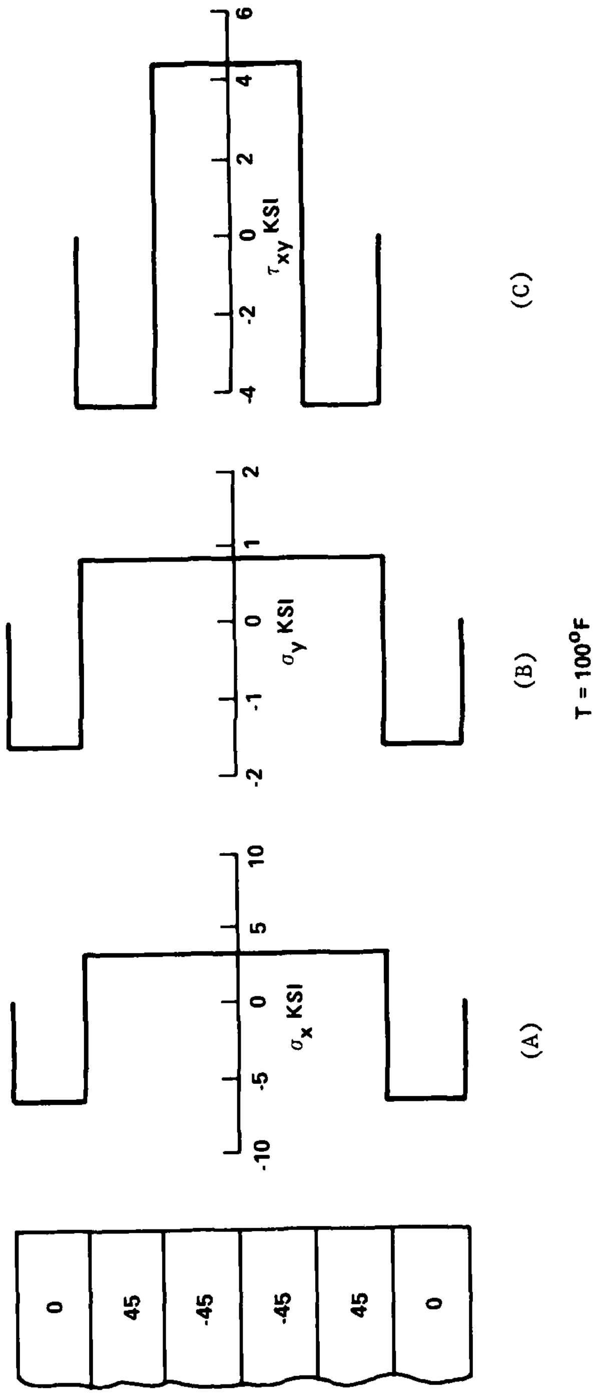


FIGURE 43. Stress Analysis for Thermal Loading as a Result of the Cure Cycle (100 series)

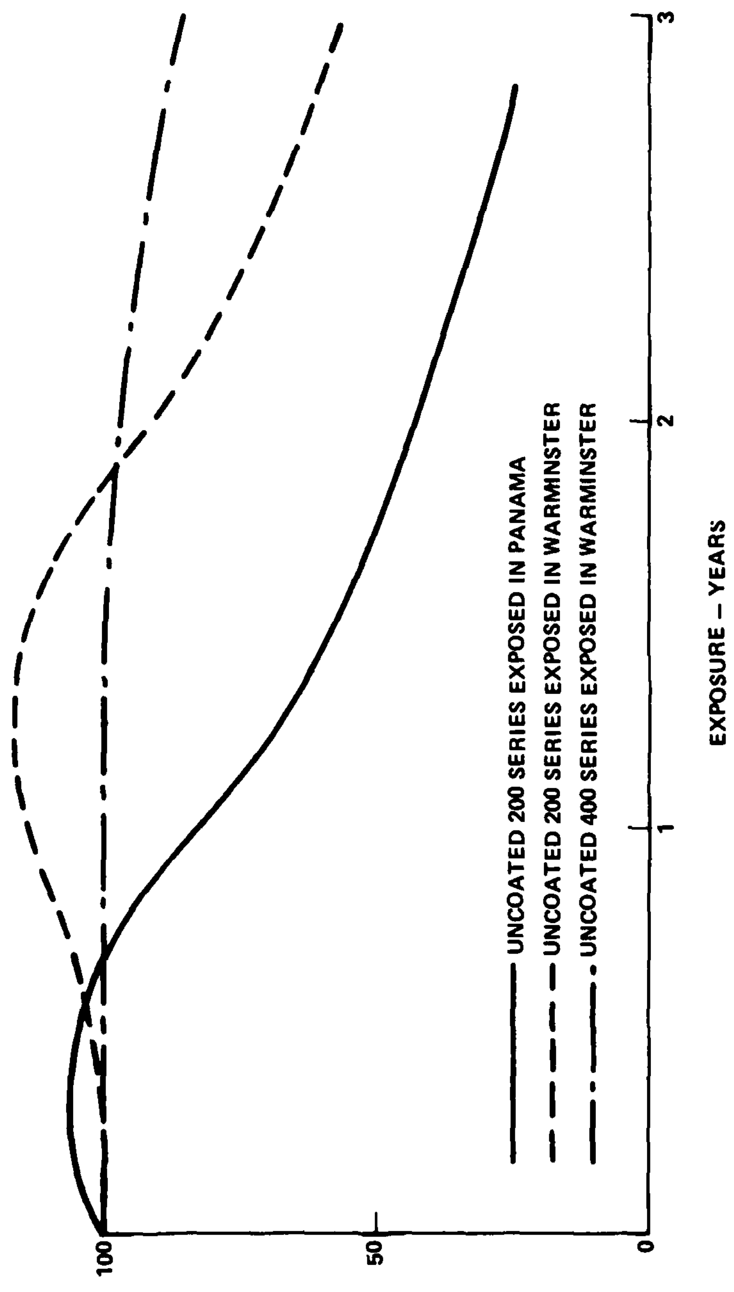


FIGURE 44. A Comparison of Typical Trends in Residual Tensile Strength as a Function of Exposure Time

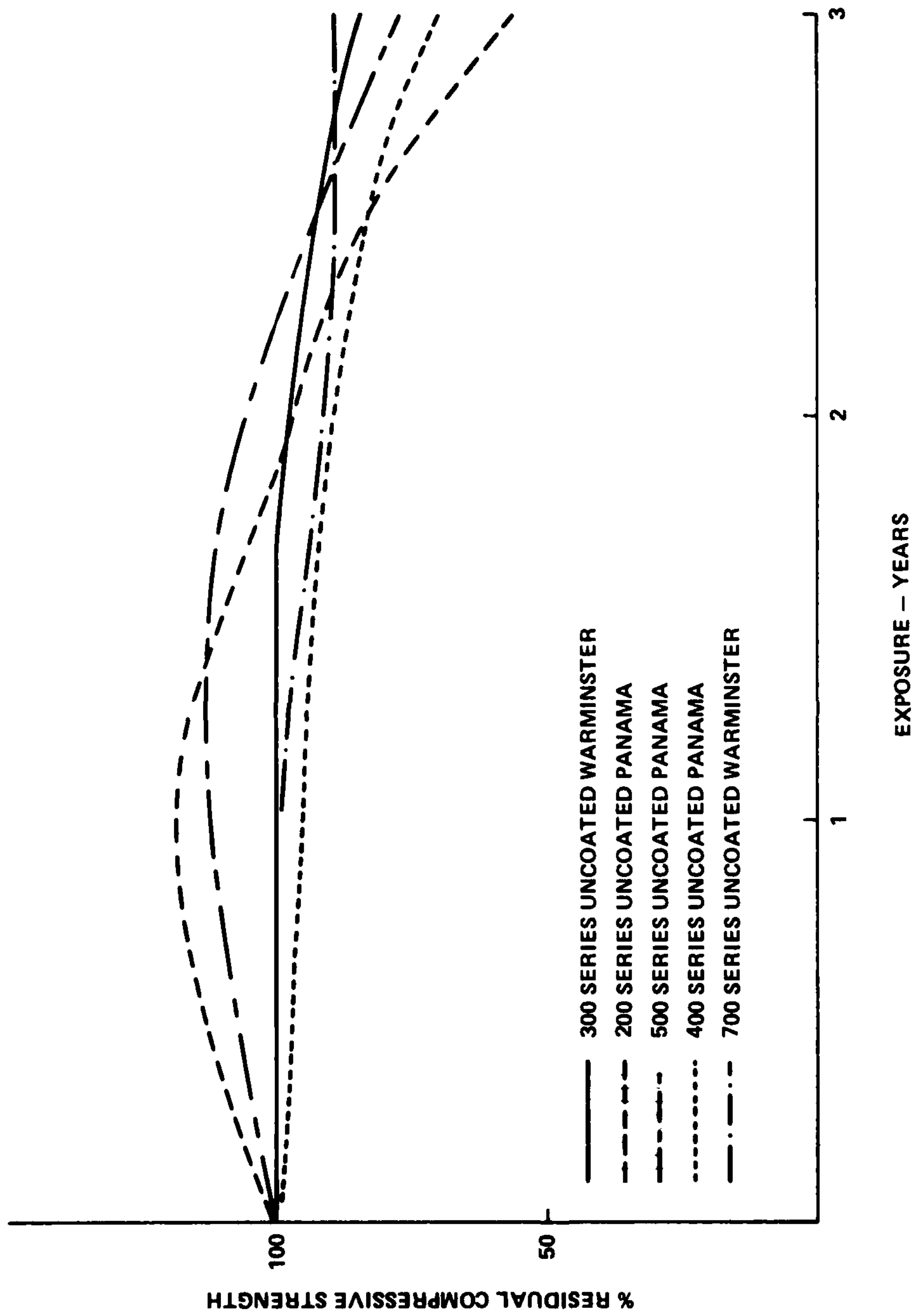


FIGURE 45 A COMPARISON OF TYPICAL TRENDS IN RESIDUAL AMBIENT COMPRESSIVE STRENGTH AS A FUNCTION OF EXPOSURE TIME

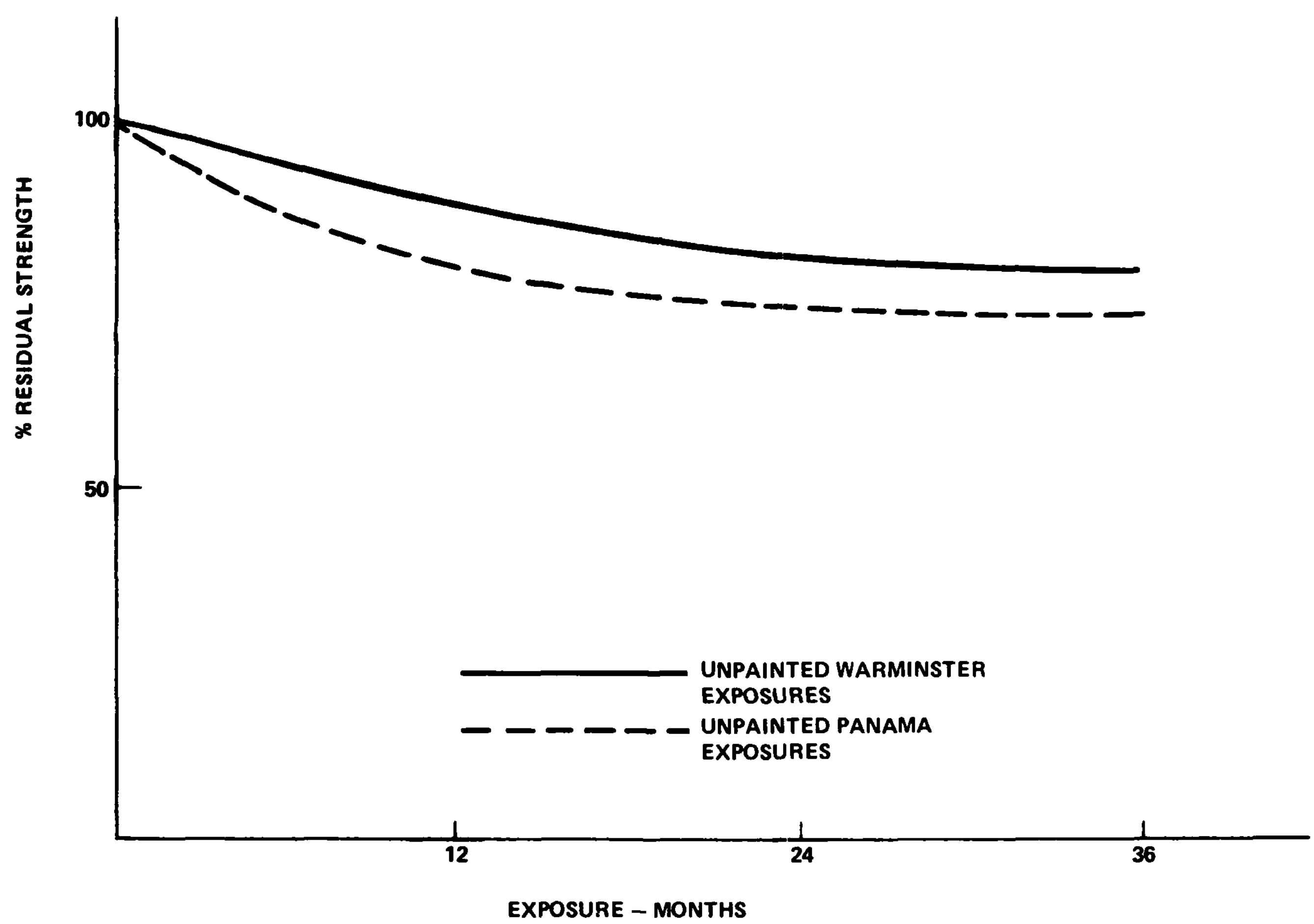


FIGURE 46. A Comparison of Typical Trends in 250° F Compressive Strength for 700 Series Material as a Function of Exposure Time

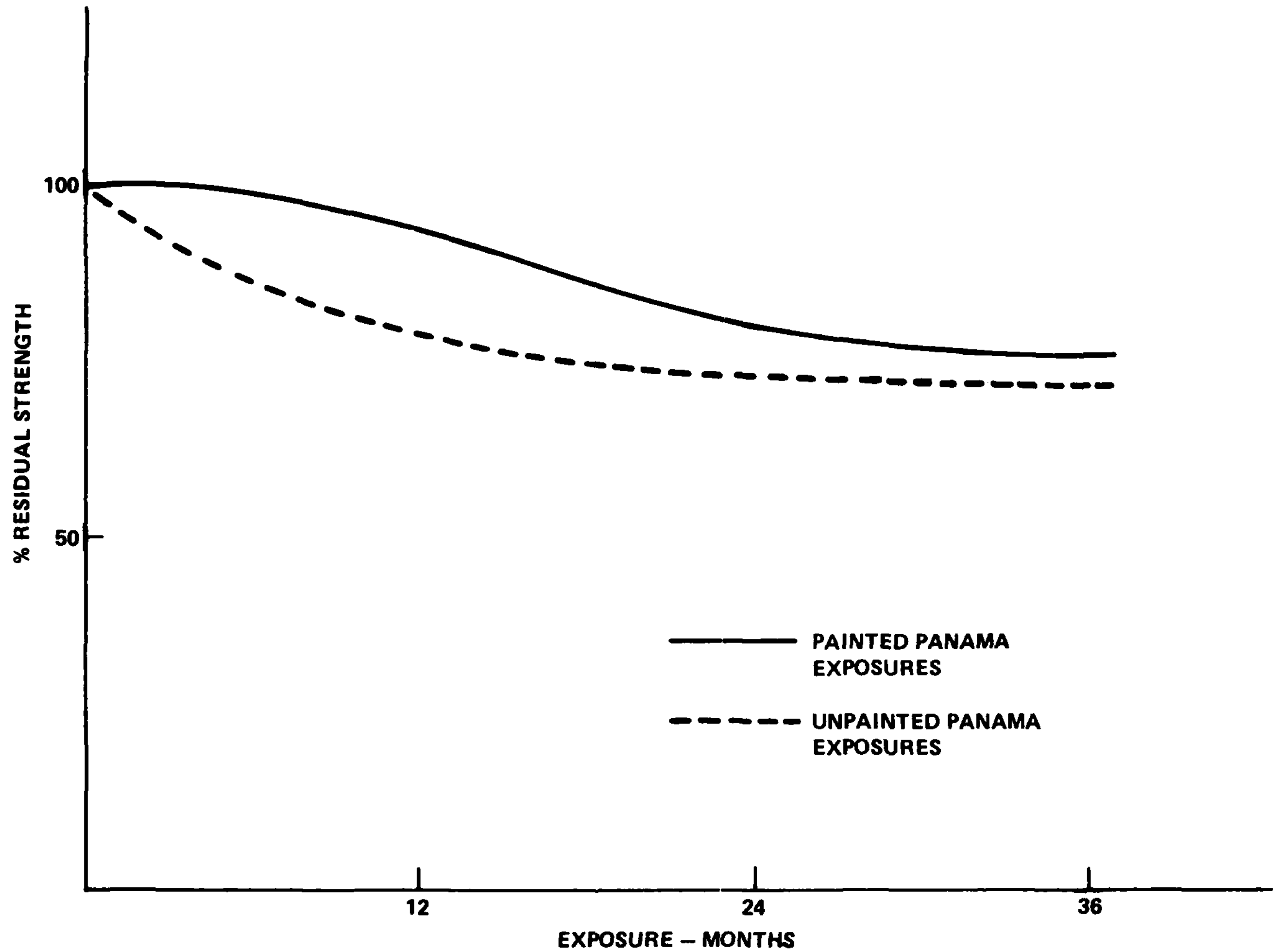


FIGURE 47 A COMPARISON OF TYPICAL TRENDS IN 250°F FLEXURE STRENGTH FOR 700 SERIES MATERIAL AS A FUNCTION OF EXPOSURE TIME

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